Do not List in B&A

A Summary of Current Program, 7/1/64

and Preliminary Report of Progress

for 7/1/63 to 6/30/64

SOUTHERN UTILIZATION RESEARCH AND

DEVELOPMENT DIVISION

of the

AGRICULTURAL RESEARCH SERVICE

UNITED STATES DEPARTMENT OF AGRICULTURE

and related work of the

STATE AGRICULTURAL EXPERIMENT STATION

This progress report of U.S.D.A. and cooperative research is primarily a tool for use of scientists and administrators in program coordination, development and evaluation; and for use of advisory committees in program review and development of recommendations for future research programs.

The summaries of progress on U.S.D.A. and cooperative research include some tentative results that have not been tested sufficiently to justify general release. Such findings, when adequately confirmed, will be released promptly through established channels. Because of this, the report is not intended for publication and should not be referred to in literature citations. Copies are distributed only to members of Department staff, advisory committee members and others having a special interest in the development of public agricultural research programs.

This report also includes a list of publications reporting results of U.S.D.A. and cooperative research issued between July 1, 1963 and June 30, 1964. Current agricultural research findings are also published in the monthly U.S.D.A. publication, Agricultural Research. This progress report was compiled in the Southern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture, New Orleans 70124, Louisiana.

UNITED STATES DEPARTMENT OF AGRICULTURE
Washington, D. C.
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INTRODUCTION

The program of the Southern Utilization Research and Development Division is an organized effort through science and technology to increase present uses and to discover and develop varied new uses for Southern farm crops. Our farmers need new markets and strengthened demand for their production. At the same time, the Nation needs the new and better products that science can create from agricultural materials. To this end the Division conducts research on cotton, cottonseed, peanuts, tung fruit, citrus and subtropical fruits, peaches, rice, sugarcane, pine gum, replacement crops, sweetpotatoes, cucumbers and other vegetables.

The Division's program includes basic and applied research in the physical and biological sciences and engineering. Basic research plays a key role in uncovering new information that may be later exploited in applied research and development. When appropriate, engineers carry out pilot-plant studies of promising laboratory developments to provide engineering and cost data essential to industrial application feasibility determinations. The Southern Division has a total staff of about 540 and the in-house scientific effort in its research program amounts to approximately 294 professional man-years. The Division consists of two Pioneering Research Laboratories (Seed Protein and Plant Fibers), eight commodity-oriented Laboratories (Cotton Finishes, Cotton Chemical Reactions, Cotton Mechanical, Cotton Physical Properties, Oilseed Crops, Food Crops, Fruit and Vegetable Products, and Naval Stores), and one Laboratory (Engineering and Development) for engineering research and development. Headquarters of the Southern Division are located at the Southern Regional Research Laboratory, New Orleans, Louisiana. The Division also has personnel and laboratory facilities at Winter Haven and Olustee, Florida; Weslaco, Texas; Raleigh, North Carolina; Houma, Louisiana; and Natick, Massachusetts.

Division scientists consult with specialists from other organizations during both the planning and the execution of the research, and cooperate actively with industry to facilitate commercialization and utilization of new findings. Much of the cooperation is informal, but some work is conducted under conditions described in written cooperative agreements and memorandums of understanding. Currently 55 such agreements are in effect.

The farm products with which the Southern Division deals not only provide food, clothing and industrial raw materials, but also contribute to the Nation's general prosperity and well-being. Cotton, the Nation's number one cash crop, has an annual farm value of about $2.5 billion. The retail value of cotton products is about $18 billion. Cottonseed, a byproduct of cotton, has a farm value of around $300 million. The retail value of its products is about $1.8 billion. Citrus grown in the U. S. has a farm value of about $500 million; vegetables over $1 billion; peanuts over $200 million;

tung about $6 million; and gum naval stores about $30 million. The retail value of refined sugar produced from sugarcane grown in the United States and Puerto Rico is about $865 million. Industries processing these agricultural crops play a vital role in the Nation's economy; agri-business today is about 30% of the Nation's total economy.

There is an urgent need to maintain the traditional food, feed and industrial outlets for agricultural products and to create new and larger markets for them. Utilization research is needed to solve existing problems, to permit adjustment to important trends and to develop entirely novel operations. The opportunities are great and, as shown by past experience, the ability of utilization research to benefit the economy is tremendous. Following are a few examples of significant developments based on the research of scientists at the Southern Division.

Solution Found for Historic Problem of Strength Losses in Wash-Wear Finishing of Cotton. Loss of strength in wash-wear cottons--a serious problem ever since all-cotton wash-wear garments were introduced--has been cut to a minimum by a new technique of mercerization developed by Department researchers. It has been discovered that pretreating cotton yarn or fabric with a mercerizing solution of caustic soda, followed by stretching, results in fabric that retains most of its strength after wash-wear chemicals are applied. The pretreatment does not affect the quality of the wash-wear finish. The new finding reverses present theories of textile chemists that substantial losses in tearing and breaking strength are inevitable in cottons given any of the wash-wear finishes now in commercial use. Cotton fabrics woven from pretreated yarn retained up to 90 percent of their breaking strength and up to 100 percent of their tearing strength after they were given wash-wear treatments. When the fabric itself was pretreated, it retained 75 to 80 percent of its tearing and breaking strength after the wash-wear finish was applied. Several textile companies are evaluating the new discovery.

Novel Milling Equipment and Techniques for Production of Deep-Milled Rice and Protein-Rich Flour. To Improve the milling of rice, ingenious laboratory equipment and techniques employing the principle of tangential abrasion have been developed by Department scientists. The method permits controlled removal and isolation of successive fractions of the surface of milled rice as a fine flour of high protein content. That the equipment causes only minimum breakage of residual kernels indicates its economic potential when it is scaled up for commercial use. The residual kernels are equivalent to conventionally milled rice in cooking tests, and have a more attractive appearance.

Extending the observations of Spanish workers engaged in P.L. 480 research, Department scientists have used the method to determine that there are higher concentrations of proteins in the peripheral layers of rice kernels. Consequently, a deep milling process offers considerable potential for the

production of a high protein rice flour which may be valuable as a food fortifier or supplement.

Commercial Scale Evaluation of Alumina Bleaching Process for Cottonseed Oil. A practical batch process for bleaching off-colored cottonseed oils with activated alumina has been developed through the pilot-plant stage. Even the most discolored oils can be upgraded considerably by removal not only of the red pigments but also--by including carbon in the alumina slurry--of the vestigial greens. Since hard-to-remove discoloration occurs in about 25% of domestic cottonseed oils, a solution to the problem is of major import to the industry. A major producer and processor of cottonseed oil has already conducted engineering cost analysis of the process and has scheduled plant-scale tests for the near future, using an economic alumina produced by a leading manufacturer. That the process also effectively achieves deodorization enhances its applicability in regaining the competitive position of cottonseed oil.

SRRL Fiber Retriever Enthusiastically Accepted by the Cotton Textile Industry. The SRRL Fiber Retriever--a simple, inexpensive Department-developed device that increases a carding machine's efficiency in removing trash from cotton to be spun--is being widely utilized in the cotton textile industry. Released to industry in the spring of 1963, the device is now being manufactured by six companies and fifteen companies have applied for licenses to manufacture it. Over 2,000 units are estimated to be in use and sales are reported to be increasing rapidly. Industry reports that the Fiber Retriever is essential for high production carding; and high production carding is a must for the cotton industry to compete with synthetics processing.

The Fiber Retriever consists of a series of baffles that replace the cleaning knives normally used in cotton carding machines. It increases cleaning efficiency at the cleaning section of the card as much as 40 percent and overall cleaning efficiency of the card as much as 12 percent. High speed production has no adverse effect on the Retriever's performance. In addition to its efficient removal of trash, the device removes a high percentage of short fibers, decreases loss of spinnable fibers, and decreases damage to the fibers. As a result, there are improvements in yarn strength and uniformity. Through use of the device processing costs are lowered and maintenance requirements for the card are decreased.

AREA NO. 1 - COTTON - BASIC AND EXPLORATORY INVESTIGATIONS

Problem. Cotton, the nation's most important fiber, is facing severe and increasing competition from synthetic fibers. Cotton is America's largest source of cash farm income and still accounts for almost two-thirds of the total U. S. mill consumption of all major fibers. However, its proportionate share of the market has been slowly decreasing as has the per capita consumption. The rapid growth of the synthetics at the expense of the natural fibers has been a phenomenon of the century. Expansion of market outlets for the chemical fibers has been based on vigorous research and development programs. The engineering and development programs of the chemical fiber industry are designed to capitalize on the special properties of each individual fiber as related to the functional use qualities desired in particular products; basically they involve the substitution of the newer fibers for cotton in cotton's traditional end use markets. Expanded research to increase the utilization potential of cotton offers the most realistic opportunity for improving cotton's competitive strength as a textile fiber and for increasing cotton consumption. Basic and exploratory investigations, studies on interrelations among fiber, yarn, and fabric properties, new and improved textile machinery, improvement of wash-wear properties and improved cotton properties and products are basic to holding existing markets or expanding the use of cotton in new applications.

Fundamental information is badly needed in applied research to help cotton gain new and maintain old markets. Fundamental knowledge of the cotton fiber as to its structure, properties, and the mechanisms involved in chemical and physical behavior serves as a basis and a guide in the design and improvement of processing machinery, mechanical and chemical processes, and in the development of new and improved cotton yarns, fabrics, finishes, and treatments. Many chemical and physical treatments, as well as textile organizations and machine designs, offer a basis for the improvement of cotton quality or lowering of processing costs. Exploratory chemical and physical research is needed to determine the true potential of such approaches prior to undertaking extensive developmental research or the construction of prototype machinery. Specific areas in which basic information is needed include the chemical properties and structure of native and modified cottons; the chemical modification of cotton cellulose; chemical reactions induced in cotton cellulose by high energy radiation; reaction mechanisms, rates, and catalysis of cotton cellulose reactions; new concepts and methods for evaluating the physical properties of native and modified cottons; relationships of the structural arrangements within cotton fibers to the physical properties of native and modified cottons; mechanisms of physical damage to cotton due to mechanical, chemical, or biological actions; fine structural changes occurring during chemical and physical modification of cotton cellulose; and correlations of the fine structure of cotton fibers with their gross behavior in textile structures.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving organic chemists, analytical chemists, physical chemists, physicists, microscopists, chemical engineers, statisticians, mathematicians, cotton technologists and textile technologists engaged in basic and exploratory studies to develop fundamental information needed in applied research to help cotton gain new and maintain old markets.

Basic research on the structure of cotton fiber and its relation to the behavior in mechanical and chemical treatments, essential to an understanding of the performance of fibers during processing and in textile products, is carried out at the Southern Regional Research Laboratory, New Orleans, Louisiana. Included is the research of the Plant Fibers Pioneering Research Laboratory to obtain basic information on the supermolecular structure of plant textile fibers; and to relate information of polymer and fiber structure to the mechanical and textile behavior of fibers. Additional basic research on chemical and physical properties and structure of cotton is being carried out: (1) under contract at Stanford Research Institute, South Pasadena, California, on determination of the structural components of the cotton fiber that contribute most to tensile strength and how they can be utilized to increase tensile and recovery properties; at Texas Agricultural Experiment Station, College Station, Texas, on the effect of variation in structure on cotton fiber properties caused by environmental and genetic factors; at the University of Tennessee, Knoxville, Tennessee, on investigations to determine the effects of fiber extensibility on fiber breakage in mechanical processing; at the Polytechnic Institute of Brooklyn, Brooklyn, N. Y., on relationship of molecular size, nature, shape, conformation, and configuration of organic non-aqueous compounds to their swelling power on cotton cellulose; and at Harris Research Laboratories, Inc., Washington, D. C., on investigation of factors influencing comfort in cotton apparel fabrics; and (2) under a grant at Massachusetts Institute of Technology, Cambridge, Massachusetts, on investigation of fiber and yarn geometry in areas of deformation in cotton fabrics.

Exploratory chemical and physical research is also conducted at New Orleans, Louisiana, as a basis for the improvement of mechanical and chemical processing, and in the development of new and improved yarns, fabrics, finishes, and treatments. Additional exploratory chemical and physical investigations are being carried out: (1) under contract at General Aniline and Film corporation, New York, N. Y., on the reaction of acetylene and related compounds with cotton cellulose; at Macrosonics Corporation, Carteret, New Jersey, on treatment of cotton fibers with acoustic energy; and at Gagliardi Research Corporation, East Greenwich, Rhode Island, on chemical modification of cotton through treatments with reagents in the vapor phase; and (2) under a grant at Textile Research Institute, Princeton, New Jersey, on crosslinking of chemically modified cotton to obtain cotton fabrics with an optimum combination of resiliency and thermoplasticity.

Other research on chemical and physical properties snd structure of cotton is in progress under grants of P.L. 480 funds to the following foreign institutions: Ministry of Commerce and Industry of State of Israel, Jerusalem, Israel, for fundamental investigation of crimp in cotton fibers and its relationship to other fiber properties (project duration - 5 yrs.); National Institute of Applied Chemical Research, Paris, France, for a fundamental study of the relation of crystallinity to accessibility in cottons (project duration - 5 yrs.); Swedish Institute for Textile Research, Goteburg, Sweden, for an investigation of setting reactions in cotton fabrics (project duration - 5 yrs.); Central Laboratory, T.N.O., Delft, Holland, for a fundamental study of the response of cotton fiber structural elements to stress (project duration - 3 yrs.); Fiber Research Institute, T.N.O., Delft, Holland, for an investigation of the fundamental mechanisms and bonding forces that could be used to improve tensile strength and other physical properties of cotton textiles (project duration - 5 yrs.); Ahmedabad Textile Industry's Research Association, Navrangpura, Ahmedabad, India, for a study of the relation between fine structure and mechanical properties of cotton fibers by swelling and stretching treatments (project duration - 5 yrs.); and for a study of the physical chemistry and thermodynamics of solution and vapor phase adsorption on and in the cotton fiber (project duration - 5 yrs.); University of Bombay, Bombay, India, for an investigation of the photochemical degradation of cotton (project duration - 5 yrs.); and for an investigation of new solvents for molecular weight determination of cellulose (project duration - 3 yrs.); Indian Central Cotton Committee, Bombay, India, for investigation of the microbial decomposition of cellulose with special reference to the effect of Indian bacterial organisms on cotton and cotton fabrics (project duration - 4 yrs.); Juan de la Cierva School of Technical Investigations, Barcelona, Spain, for a study of the measurement of "total hairiness" of cotton yarn and the determination of mechanical factors contributing toward its formation (project duration - 5 yrs.); and The Cotton Silk and Man-Made Fibres Research Association, Shirley Institute, Didsbury, Manchester, England, for a study of the effect of swelling agents on the fine structure of cotton (project duration - 5 yrs.).

Exploratory chemical and physical investigations are in progress under grants of P.L. 480 funds to the following foreign institutions: Birkbeck College of University of London, London, England, for a fundamental study of the preparation and properties of phosphazene and phosphoryl chloride derivatives having potential for reaction with cotton cellulose (project duration - 4 yrs.); Indian Central Cotton Committee, Bombay, India, for an investigation of the preparation of radioresistant and radiosensitive celluloses (project duration - 5 yrs.); Ministry of Commerce and Industry of State of Israel, Jerusalem, Israel, for a fundamental study of the oxidation of cotton and crosslinked cotton by various oxidizing agents (project duration - 3 yrs.); and Chalmers University of Technology, Gothenburg, Sweden, for a basic investigation of the behavior of cotton subjected to aerodynamic forces (project duration - 3 yrs.).

The Federal in-house scientific effort devoted to research in this area totals 63.9 professional man-years. Of this number 36.6 is devoted to chemical and physical properties and structure and 27.3 to exploratory chemical and physical investigations. The domestic contract and grant research involves an additional 13.4 man-years, 8.0 being on chemical and physical properties and structure, and 5.4 on exploratory chemical and physical investigations. P.L. 480 research involves 16 grants, of which 12 are on chemical and physical properties and structure and 4 on exploratory chemical and physical investigations.

The following lines of work were terminated during the year: (1) Evaluation of the swelling behavior of cotton fibers in various environments by observation of the untwisting of single yarns (under chemical and physical properties and structure); and (2) Determination of the mechanics of nep formation in cotton during textile mechanical processing; (3) A fundamental investigation of the drying of chemically modified cotton; (4) Fundamental investigation of preparation and properties of esters, anhydrides, hydrazides, pseudohalides, fluorides, and related compounds of the phosphonitrilic chlorides for use in preparing new cotton products (P.L. 480 project); and (5) Fundamental study of the pyrolysis of cotton cellulose (P.L. 480 project), (under exploratory chemical and physical investigations).

PROGRAM OF STATE EXPERIMENT STATIONS

Station research on cotton and cotton fiber utilization is not extensive. The Tennessee station carries out a program of research designed to evaluate cotton fiber properties for the plant breeder. In this laboratory, instruments have been developed for measuring length, fineness, maturity, tenacity, elongation, crimp, compressibility and other properties of fibers. Texas workers are evaluating the effect of seed cotton moisture content on cotton fiber quality and the effects of mechanical harvesting of cotton. Another study attempts to determine the relative usefulness of selected fiber properties and selective objective measures of quality in predicting spinning performance.

In a study directed toward development of new principles and techniques for ginning cotton, the characteristics and properties of seed cotton lint and seed which are related to basic ginning processes are being investigated. Work directed to development of new and improved engineering principles for maintaining cotton quality involves application of chemicals and other spindle moistening materials during the harvesting process and determination of effects on spinning performance.

The Arizona station is studying cross linking of cotton to produce wash and wear type of fabric and the Texas station is evaluating selected cotton wash and wear fabrics in use tests.

Four regional studies are directed to: (1) determining the relation of fiber properties to end-product performance; (2) determining the mechanism

of fabric stress absorption and performance; (3) determining properties of textile-furnishing fabrics and their importance to consumer satisfaction; and (4) determining the effects of atmospheric conditions on fabrics. Another study relates to evaluation of the effects of radiation on the physical and chemical properties of the fibers of selected fabric.

A total of 13.1 professional man-years is devoted to this research.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical and Physical Properties and Structure

1. Fundamental Investigations of Adsorption and Swelling Phenomena in Native and Modified Cottons. Research to evaluate the swelling behavior of cotton fibers in various environments by observation of the untwisting of single yarns has been completed. Estimates of the relative lateral swelling of cotton fibers at room temperature in 100 different liquids were made by observation of the extent of untwisting of singles yarns during the first three minutes of immersion in the liquids. Highest values were for the known intracrystalline swelling agents; intermediate values were grouped about the value for distilled water; and values for most nonaqueous liquids were distributed exponentially from zero. In general, results of swelling evaluations by microscopical techniques agreed with ranking by the untwisting technique. Nonaqueous liquids merit more complete study. The untwisting technique provides a rapid and simple method of screening liquids with respect to their swelling characteristics; it should prove useful in screening and classifying reagents employed in the wet processing of cotton textiles. (S2 1-182).

Microscopical investigations of absorption and swelling phenomena in native, mercerized and modified cottons are in progress. Stains containing heavy metals are being explored for their possible use in the research. Ultrathin sections of raw cotton, exposed on the specimen grid of the electron microscope to solutions of phosphotungstic acid, of uranyl acetate, and of lead hydroxide, all showed heavy deposition of the metal in the primary wall area of the section when observed at a magnification of approximately 100,000 X and photographed at a magnification of approximately 50,000 X, but no quantitative interpretation was possible. Whole fibers, soaked in solutions of the stains, are being examined by cross section to evaluate extents of penetration and deposition by this approach. Based on recent and earlier experiments, it would appear that chromium and lead salts are preferable stains for following changes in the fine structure of cotton microscopically. Samples of cotton freeze-dried from various swelling agents are being compared using the electron microscope. Initial experiments have shown that freeze-drying directly from 18% sodium hydroxide solution apparently badly degrades the cotton fibers, whereas washing out the alkali prior to freeze-drying gave samples having the familiar characteristics of mercerized cotton fibers. (S2 1-209).

Research has been initiated to determine the properties and structural characteristics of cotton fibers which influence the capacity of the fibers to sorb alkaline solution, and thus to increase the usefulness of the alkali swelling centrifuge (AC) test for characterizing cottons, both native and after chemical and/or mechanical treatments. Examination of the procedural details of the AC test as applied to two cottons differing considerably in physical properties is in progress as the first investigative phase of the research. Previous work has suggested that the AC test might be a promising method to investigate swelling response of chemically treated cotton fibers and, in particular, to indicate damage to the cotton fiber primary wall. (S2 1-249, Pending).

2. Basic Studies of the Relationships of the Structural Arrangements Within Cotton Fibers to the Physical Properties of Native and Modified Cottons. Electron microscopical observations of cotton fragments treated on the specimen grid continue to reveal details of structural changes bearing on the mechanism of reactions within the cotton fiber. The effect of crosslinking on substituted cotton (investigated by dimethylol ethyleneurea treatment of acetylated, benzylated and cyanoethylated samples of different degrees of substitution) was, in general, that crosslinking increased susceptibility of lamellar fragments to hydrolytic degradation. Surface replicas of cyanoethylated cotton indicated that upon heating and stretching, normal surface rugosities are largely lost; the internal structures of highly substituted samples subsequently heated to 175° C were similar to those of unheated samples. Electron micrographs of fragments of untreated native cotton exposed on the grid to several intra-crystalline swelling agents indicated that, in thinly divided specimens, swelling by reagents of this class results in complete disruption of the fibrillar aggregates to form small discrete particles; this implies that the more easily accessible portions of the fibrillar mass are dissolved to leave the more well-ordered fractions free of any formal association one with another.

Ultrathin sections of untreated cotton samples, when prepared by the deceresol-methanol-methacrylate expansion technique, produced cross sections in which the cell wall was separated in many layers concentric about the lumen. In both slack- and tension-mercerized samples, concentricity was lost, and the expanded fiber section appeared to have a "honey-combed" structure. Samples slack- or tension-mercerized and subsequently crosslinked with formaldehyde had compact cross sections which appeared solid throughout even after application of the layer-expansion technique. Dimethylol ethyleneurea-treated samples exhibited wide variability in behavior. Postmercerization after crosslinking with formaldehyde had the effect of opening up fiber structure. When effects of postmercerization were investigated on samples crosslinked by butadiene diepoxide, no layer separation could be induced, but surprisingly, postmercerization dramatically lowered the refractive index of cottons crosslinked by this finishing agent. Observations of ultrathin sections taken from fibers treated with acrylonitrile after gamma irradiation of the cotton indicated heavy reaction in the outer layers of the fiber only. (S2 1-174).

In further research on the effect of gross and fine structure on the physical behavior of cotton fibers, a method has been developed for preventing cotton fibers from collapsing upon drying which should prove useful in studies of the fiber structure produced by controlled conditions of growth. It has been established that wetting the fibers with sucrose, potassium carbonate or potassium phosphate solutions is most satisfactory for maintaining them in the cylindrical shape as found in bolls prior to drying. Also, after several rewettings with sucrose solution and dryings, the fibrillar structure becomes more apparent. Another finding is that fiber crosslinked (as for example by treatment with formaldehyde) prior to initial drying will collapse on drying but will return to essentially cylindrical shape on rewetting with water. Work is in progress to study the relationships of fiber structure to physical behaviors employing cottons selected for unusual properties. In recent work on Asiatic cottons grown in both India and the U. S., and another group of breeder's strain cottons selected for unusually high strengths or elongations, a four-fold difference in tensile modulus and a two-fold difference in toughness were found. The largest range was found among the cottons chosen for high strengths. Differences in cellulose density were within the range found for other cottons even though wide ranges in other properties exist. The wide range in mechanical properties found among the breeders samples is evidence that cottons of widely different characteristics can be produced by breeding. Effort in future work will be concentrated on specially grown cottons with structure altered by known growth variables. (S2 1-208).

Basic research is in progress under contract at Texas Agricultural Experiment Station on the effect of variation in structure on cotton fiber properties caused by environmental and genetic factors. In this research, cottons will be grown in growth chambers under different conditions of illumination, temperature, humidity, etc. to produce samples having differences in fiber structure. The first series of cottons grown under controlled conditions (continuous light, 85° F., 70% relative humidity) have been harvested. Considerable information on differential response of varieties to controlled growth was obtained with this series of plants. Closely controlled conditions apparently cause plant growth unbalances and shedding of flowers. Mechanical difficulties with the growth chambers which caused delays in the research have now been remedied. (S2 1-217(C)).

Initial contract research at Stanford Research Institute to determine fiber components that contribute most to strength properties of cotton has involved a systematic investigation of the effects of mercerization and physical manipulations during the process on yarn strength. Yarns treated in 18% or 24% sodium hydroxide under comparable conditions had essentially identical properties but yarns treated with 10% caustic were changed very slightly and more nearly resembled unmercerized yarn. Strongest yarns were produced when the highest tensions were applied. These were least extensible. Changing the temperature at which mercerization was carried out had little effect on yarn properties. Yarns were also swollen in nine other swelling agents with measurements made of shrinkage rates and determination

of yarn strengths. Techniques will be developed for conducting electron microscope studies on surface replicas of cotton fibers and on microfibrils to formulate theory explaining the changes in tensile properties of yarn and fibers that result from swelling treatments. (S2 1-206(C)).

Fundamental studies of the role played by the structural elements of the cotton fiber in response to stress are being conducted in P.L. 480 research at the Central Laboratory, T.N.O. Through the use of modern microtechniques for manipulating and observing single fibers, a better understanding is being obtained of the internal movements that occur within the cotton fiber while it is being subjected to torsion and stretching. Fibers treated by resin treatments commonly used in wash-wear finishing of cotton have been found to be more rigid to torsion and to begin to form cracks and break at lower torsion than untreated fibers. Progress is being made in obtaining basic knowledge of cotton fibers that eventually will be directed toward efforts to improve cotton fiber properties through cotton breeding programs and improvements in cotton processing. (UR-E19-(20)-4).

In P.L. 480 research at the Swedish Institute for Textile Research, reactions which will cause setting in cotton fabrics and garments are being investigated. Treatment of cotton fabrics with solutions of certain inexpensive alkalis or inorganic salts which cause swelling of the cotton fibers has been shown to cause the relaxation of internal stresses in the fabrics. This treatment, which is generally known as "setting," decreases surface mussiness of the fabric, and in combination with standard resin treatments, results in improved wash-wear properties. In recent developments, it has been observed that the conditions under which deswelling of the fibers occur during treatment greatly influence the "setting" effect. Progress is being made toward providing the basis for reducing the amount of resin required to provide acceptable wash-wear qualities in cotton textiles. (UR-E26-(20)-2).

3. Elucidation of Mechanisms of Physical Damage to Cotton Due to Mechanical Chemical, Physical or Biological Actions. A P.L. 480 project is underway at the Technological Laboratory of the Indian Central Cotton Committee in Bombay, India, to investigate the degradation of cotton cellulose by bacteria indigenous to the Bombay area. Progress is being made in the collection and isolation of bacterial and fungal species suspected of causing damage to cotton and cotton products in this region. Cultures of cellulolytic organisms known to exist in the U. S. have been furnished to the Principal Investigator for comparative studies. Work is underway to determine the mode of attack and the mechanism of degradation of cotton cellulose by the various microorganisms isolated in the study. It is expected that the information obtained in the research will be useful in devising means to minimize or prevent microbiological damage to cotton and cotton products which is estimated to cause losses running close to a billion dollars annually to the U. S. textile industry and to users of cotton products. (UR-A7-(20)-32).

An investigation of the photochemical breakdown of cotton under different conditions of exposure to radiation is being continued in P.L. 480 research in India at the University of Bombay. It is well known that cotton fabrics are weakened by prolonged exposure to sunlight or to strong illumination. Progress is being made toward determining the mechanisms by which photosensitization and photolytic degradation of cotton and selected modified cottons take place. Basic knowledge of the reaction mechanism and kinetics is expected to be useful in devising practical means to prevent the deterioration of exposed cotton fabrics by means of chemical inhibitors or screening agents that prevent or interfere with the sequence of reactions involved (UR-A7-(20)-4).

Under a P.L. 480 grant at the University of Bombay, a study is being made of new, more stable solvent systems for cellulose in the determination of the average molecular weight of cellulose by the disperse viscosity technique. Copper complex solvent systems widely used for this purpose are extremely oxygen-sensitive, a factor which greatly complicates their preparation, storage, and use. Substantial progress is being made in studies of several iron tartrate complex solvents that are relatively insensitive to atmospheric oxygen, and in relating data obtained with them to comparable data with the older copper complex systems. Means have been developed to apply a two-component solvent system to the dissolution of high degree of polymerization cottons and mercerized samples that are difficult to dissolve in the usual solvents, thus permitting satisfactory measurement of these materials. The information obtained in this project will be useful in following the degradation of cotton by various treatments, through the application of a simpler procedure for intrinsic viscosity measurement. (UR-A7-(20)-30).

4. Investigation of the Structural and Compositional Changes Occurring During Chemical and Physical Modification of Cotton Cellulose. Work on fine structure of plant fibers has been pursued along a number of lines by the Plant Fibers Pioneering Research Laboratory. A new technique, heterogeneous reaction kinetics, has been employed in an effort to throw new light on the nature of cellulose structure. As is well-known, the rate of chemical reaction in a heterogeneous system is dependent on the structure of the system. Cellulose is generally held to be a two-phase system consisting of crystalline and noncrystalline (amorphous) fractions. These in turn are related to the microfibrillar organization of the cellulose.

In view of the strong influence, apparently, of structure and pretreatment of cellulose on reaction rates as well as observation of two consecutively differing rates of acetylation, cyanoethylation, and ester saponification, it was concluded that heterogeneous reaction kinetics of cellulose might be employed to throw considerable light on the nature of the cellulose structure and perhaps give information on its variability or uniformity in plant fibers.

The reaction of cellulose with acrylonitrile (cyanoethylation) under

alkaline conditions has been studied quite extensively during the past year, though this has been supplemented somewhat in recent months with propionic and furoic acid esterification in pyridine.

In the cyanoethylation reaction the temperature has proved to be an important factor affecting the position of the abrupt change of rate (intersection of the two rate curves) during the progress of the reaction. Reduction of the crystallinity of the cellulose by 25% brought about by ethylenediamine treatments caused a decrease in the break point in the rate curves at 50° C. only from DS = 2.2 to 2.0. It was established by X-ray diffraction analysis that the break point in the rate curve is closely associated with the loss of crystalline structure of the cellulose.

While a great deal of information has thus been obtained with the cyanoethylation reaction, this has not proved to be an entirely suitable reaction. Dilution of acrylonitrile with four parts to one of pyridine, xylene, toluene, benzene, or carbon tetrachloride, which greatly reduced the rate of reaction, had very little influence on the position of intersection of the rate curves, but favored side reactions.

The most serious side reactions were those becoming prominent during the latter stages of reaction. The effects of these side reactions upon the kinetic interpretation is such as to suggest the advisability of exploring other reactions less influenced by these problems. The esterification of cellulose in propionic acid in pyridine gives considerable promise of such a reaction at the present time.

During the year a study was made of mature cotton fibers taken from unopened cotton bolls. The objective was to throw light on the controversy as to whether cotton cellulose is already crystalline in the moist state of the fibers in unopen bolls, or alternatively, crystallizes at the time the boll opens and the fibers dry out. In these studies the bolls were opened and sampled and the fibers preserved in various ways. The crystallinity of the cellulose in the undried fibers was studied by X-ray diffractometer methods. The crystallinity of portions of the fibers was compared with that of portions either dried directly or from which the water has been removed in various "indirect" ways, designed to preserve the structure existing before drying. The results lead to the conclusions that crystalline cellulose is already present in the never-dried but mature cotton fiber. However, although crystallinity was always found in the cellulose of undried fibers, the difference in crystallinity observed between directly and indirectly dried fibers leads to the conclusion that additional crystallization of the cellulose must take place during initial drying of the water-wet fiber. Crystallization evidently continues for some time after the initial drying, as indicated by the very high crystallinity of fiber which has undergone a period of storage.

When indirectly dried fiber (e.g., freeze-dried) was rewetted in water, an increase in the degree of crystallinity was observed, even above that of

fiber dried directly from the boll. The preservation of certain amounts of amorphous cellulose by the indirect drying methods (solvent-exchange, freeze-drying, drying above the critical temperature and crosslinking before drying) can be explained by assuming that additional crystallization takes place subsequently upon drying of the rewetted fiber. It was supported by observation of lower density and higher dye accessibility in samples dried by these indirect methods. The results of this study are being supplemented by studies of fibers removed from unopened cotton bolls and various earlier stages of growth from 20 days after flowering to maturity.

In continuing studies on the effect of formic acid upon the crystal lattice transformation of cellulose triacetate in highly acetylated cotton fiber it was concluded that the more concentrated formic acid solutions tended to swell or dissolve cellulose triacetate, thus promoting the polymorphic transformation.

Finally, excellent progress is being made on the effects of selected chemical modification on the structure and physical properties of cotton fiber. With the perfection of techniques for the preparation of reacted fibers it is expected that research in this area will greatly accelerate during the coming year.

In other in-house research (not in PF Pioneering Research Laboratory), investigations of improved infrared spectral techniques for the study of modified cottons to evaluate molecular structural changes produced by chemical and physical treatments have continued. Differential infrared spectra of crystalline modifications I, II, III, and IV of cellulose in which the decrystallized form of each modification was used as the reference shows that these four modifications may be distinguished by this method; conventional infrared spectra have not shown important differences. The interpretation of the differential spectra will be continued to arrive at probable frequency assignments. It also has proven possible to distinguish cottons treated with the dimethylol derivatives of methyl-, ethyl-, and isopropyl carbamates for wash-wear properties by means of the spectra of the acid hydrolyzates of these materials. Characterization of the infrared spectra associated with the complex molecular architecture of physically and chemically modified cotton cellulose is an important step in gaining insight into the structure and reactions of improved cotton textile materials. (S2 1-220).

Research was initiated to develop improved instrumental techniques for elemental analysis of additively and chemically modified cottons. Satisfactory quantitative analysis of a wide variety of cotton textile materials for the following elements by X-ray fluorescence techniques has been achieved: lead, mercury, tellurium, antimony, tin, cadmium, zirconium, bromine, selenium, zinc, copper, cobalt, iron, chromium, titanium, calcium, chlorine, sulfur, phosphorus, silicon, and aluminum. The X-ray method is generally much faster and of equal precision as the standard "wet" methods of analysis for these elements. The extension of the method to elements of lower atomic number will require radical changes in instrumental design.

In a limited number of cases, anomalous results have been obtained with the X-ray fluorescence techniques when the elemental matrix is of differing particle size. This will be investigated further. Another instrumental technique--atomic adsorption spectroscopy--appears to have definite value in the analysis of a limited number of elements, such as sodium, zinc, copper, and cadmium at the part-per-million level. A survey of the scope and limitations of these two techniques is being continued. (S2 1-218).

In further work on the separation and identification of the cleavage products of partially etherified cottons to elucidate the structure of the modified cottons, techniques for both hydrolysis and oxidation of modified cottons have been developed by which cleavage products can be prepared for identification purposes. Due to difficulties encountered in isolating stable hydrolysis products from aminoethylated cotton, emphasis was shifted to oxidation of formaldehyde treated cottons and hydrolysis of sulfone treated cottons. It has been found that samples of three types of formaldehyde treated cotton fabrics (Form W, D, and V processes) at the same level of formaldehyde content vary in rate and degree of oxidation with sodium periodate depending on method of formaldehyde treatment. Although this study did not lead to isolation of cleavage products as expected, it did result in the discovery of differences in accessibility of the three types of formaldehyde modified cottons. Electron microscopy and X-ray diffraction measurements both indicate that each of the three types of formaldehyde treated cottons are crosslinked but the Form V type is different from the others. In initial work on hydrolysis of methyl vinyl sulfone-treated fabrics, a sulfone-substituted glucose fraction has been separated from the hydrolyzate by use of gas chromotography. Known sulfone adducts of glucose have been prepared for comparison with the hydrolysis products. Elucidation of the chemical structure of the various etherified cottons will aid in the development of reactions designed to yield modified cottons having specific and controlled properties. (S2 1-214).

A fundamental investigation of the effect of swelling and stretching treatments on the fine structure and mechanical properties of cotton fibers is being conducted under a P.L. 480 grant at the Amedabad Textile Industry's Research Association (ATIRA), in India. The effect on fiber fine structure, as revealed by X-ray, microscopic and modulus measurements, of swelling fibers under tension with agents such as solutions of sodium hydroxide, ethylene diamine, and zinc chloride is under study. It has been shown in early stages of the research that orientation is much more decisive than crystallinity in determining the elastic modulus. The information obtained in the project is expected to be useful in the selection of treatments to improve the mechanical behavior of cotton products. (UR-A7-(20)-19).

In P.L. 480 research being conducted in Paris, France at the National Institute of Applied Chemical Research, a basic study of the fine structure of the cotton fiber is being made in an effort to relate the fine structure to other fiber properties that are important in the processing and use of cotton. Improved methods, both physical and chemical, have been devised for

measuring differences in the fine structure of cottons. These have been applied to a typical U. S. cotton of Deltapine variety, the fiber properties of which have been extensively studied in several laboratories, and are thoroughly known. These studies are now being extended to a series of raw, purified and chemically crosslinked cotton yarns, all spun from the same Deltapine cotton. Three commonly used crosslinking treatments, all easily measured by analytical procedures, are included in this phase of the investigation. The information obtained eventually will be translated into the development of improved cotton products. (UR-E9-(20)-61).

5. Relationship of Cotton Fiber Gross Structure to Behavior of the Fibers in Textile Structures. Fundamental investigations of the interfiber frictional force and associated fiber properties of cotton have continued. Experiments to determine the effect of drafting direction on fiber hook removal, and on processing performance, for a short staple cotton showed that the optimum draft procedure to minimize fiber hooks was not the same as previously found for a medium staple cotton. It was ascertained that the uniformity of sliver and roving was better when the "majority" hooks were drafted in the trailing position at each process. The amount of hooks entering spinning affected end breakage greater when spinning fine yarns. In experiments with a medium staple cotton, the amount of fibers (fiber mass) entering the drawing frame and removal of fiber hooks were found to be inversely related. Therefore, by using a light weight sliver (approximately 50 grains/yard) entering drawing, mills will not only obtain better uniformity but also more effective hook removal, which in turn will increase spinning performance and permit the use of higher spinning speeds. The effects of fiber hook removal and draft direction on processing performance for an extra long staple cotton is now being studied. In another phase of work, an inverse relationship between fiber bundle strength and friction was found in experiments on selected cottons differing appreciably in fiber strength. This indicates that the previously observed relationship between fiber bundle strength and drafting tenacity was not due to surface properties. (S2 1-201).

A fundamental investigation of fiber crimp, a property possibly responsible for differences in mechanical processing behavior of cotton fibers, is now in its final phases under a P.L. 480 grant at the Ministry of Commerce and Industry of the State of Israel. An optical projection system was developed to measure crimp in two perpendicular planes. The apparatus has now been improved through the development of a special curve tracer in conjunction with an electronic computer that permits the continuous and dynamic measurement of the crimp diameter, which is considered to be the main crimp parameter of cotton fibers. Crimp diameter decreased with tension for Deltapine 15 fibers. When energy required to uncrimp fibers was measured, that for Deltapine fibers remained essentially constant after the first stretching cycle. Energy for Acala 1517 fibers decreased with the first through fourth stretching cycles and increased with period of relaxation, a distinct difference in behavior of these two cottons. These improved techniques for evaluating fiber crimp are being applied to a number of cottons of differing

physical characteristics on which extensive fiber property data are available. (UR-A10-(20)-5).

B. Exploratory Chemical and Physical Investigations

1. Exploratory Chemical Modification of Cotton Cellulose. Seven classes of widely used polymers (polyvinyl esters, polyvinyl ethers, polyvinyl acetals, alkyl polyacrylates, methyl hydrogen polysiloxanes, dimethyl siloxanes, and linear polyesters) have been successfully crosslinked as coatings on cotton by rapid curing with free-radical initiators. The coatings produced are highly durable. Increased wet and dry wrinkle recovery were obtained using only moderate add-ons of crosslinked silicones (dimethyl silicone, or methyl hydrogen polysiloxane). High water repellency and fair flex abrasion resistance were obtainable in these treatments. In another phase of work, the sulfone method of wash-wear finishing of cotton has been greatly improved. High crease recovery has been obtained in very short curing times (25-35 seconds) in applying bis(hydroxyethyl)sulfone at 175-185° C. The high wrinkle resistance is achieved without excessive strength losses, and means have been found for obviating the need for an afterbleach without the lowering of crease recovery usually caused by yellowing preventatives. Emphasis in a new replacement project will be on improvement of abrasion resistance in wash-wear cottons. (S2 1-186).

Basic research is in progress on the crosslinking of various physically modified crystalline forms of cotton as a means of producing resilient cotton textiles having improved appearance and durability to wearing. The strength of crosslinked cotton was found to depend mainly on its physical state prior to crosslinking, and slightly on the crosslinking agent used. Practically unchanged breaking and tearing strength can be obtained together with high wrinkle resistance, contrary to current beliefs. This was initially accomplished by processes involving slack mercerization and restretching of cotton yarn in alkali, weaving this into fabric, scouring the fabric, and crosslinking with such wash-wear agents as formaldehyde, DMEU, APO, or bis(hydroxyethyl)sulfone. Similar results were also achieved when the scouring step in the process was omitted. Extensibility in the crosslinked special fabrics was unexpectedly high, and the degree of orientation of the cellulose crystallites was unexpectedly low, considering the recrystallizing and reorienting treatments used. The finding that strength losses once thought to be unavoidable in wash-wear finishing of cotton textiles can be eliminated is leading to evaluation of the new process by industry. (S2 1-210).

In research to develop improved methods of etherifying cotton cellulose, the catalytic effect of alkali metal iodides on the etherification of cellulose with benzyl chloride, discovered in previous work, has proven applicable to etherification with other types of organic chlorides. New cellulose ethers have been prepared using 1,3-dichloropropene, 1,4-bis(chloromethyl) benzene, 2,4-dichlorobutene, 1,4-dichloro-2-butyne, and 1,3-dichloropropane. The latter four treating agents are commercially available. The reaction of

suitably activated cotton yarn with the chloro compounds has given a high degree of cellulose substitution and crosslinking with high elongation and little strength loss. (1-Naphthyl)methyl cellulose at a degree of substitution of 0.76 showed considerable thermoplasticity, but the false-twisted yarn heat-set less readily than benzyl cellulose. In other work, a rapid and potentially inexpensive method of phosphorylating cotton has been developed with the aim of reacting this material with alcohols to form etherified cottons. The new phosphorylation method produces fabrics having higher breaking strength than those made by conventional processes such as the urea-phosphate method. (S2 1-219).

The research on exploration of cellulosic crosslinks capable of being broken and reformed at will has been terminated. Further work has shown that the reduced disulfide crosslinks in acethydrazide disulfide-treated dialdehyde cotton tend to reform spontaneously by air oxidation. The reformation of these crosslinks has also been achieved by wet state oxidation. High wet wrinkle recoveries were developed in these samples by oxidation in air-saturated water. The background information on "reversible crosslinks" from these investigations may form the basis for development of a new type of wash-and-wear finish in which creases and pleats may be introduced or relocated during fabrication or alteration of cotton garments. Investigations of spatial and structural effects of reversible and conventional crosslinks in cotton will be undertaken under a new project. (S2 1-168).

A fundamental study of reactions between epoxy compounds or their halohydrin precursors and cotton cellulose is in progress. Continued progress has been made in elucidation of the mechanisms of the cellulose-epichlorohydrin and cellulose-butadiene diepoxide reactions, which can result in both dry and wet crease resistant cottons. A comparison of these mechanisms with those involved in the reaction of cotton with halohydrin precursors of butadiene diepoxide and epichlorohydrin (principally 1,3-dichloropropanol-2, which results in cottons possessing only wet crease resistance) should make it possible to control the reactions between cotton and these compounds to produce the desired effects efficiently. Data are being obtained to explain differences in the cotton-epichlorohydrin reactions in the presence and absence of salts; and titration curves of cellulosic products resulting from reactions between alkali cellulose, epichlorohydrin, and tertiary amines have been determined and are being used to elucidate cellulose-epichlorohydrin reaction mechanisms. (S2 1-216).

The contractor (General Aniline and Film Corporation) has developed suitable methods for the vinylation of cotton yarns and fabrics by reaction with acetylene. Attempts to vinylate cotton with other reagents gave low apparent vinyl ether contents. Using acetylene, degrees of substitution as high as 0.53 have been obtained. Predominantly the vinyl ether of cellulose is formed, with some attendant formation of acetal crosslinks of the cotton as evidenced by insolubility in Cuene. Only moderate changes in textile properties have been observed to result from vinylation; however, further chemical reactions conducted at the vinyl group may lead to a variety of

interesting modified cottons with potential commercial utility. (S2 1-199(C)).

Research to gain fundamental knowledge of the influence of lead compounds on cotton, and to impart useful properties to cotton through application of new finishing agents based upon lead and other metal compounds has continued in cooperation with the International Lead and Zinc Research Organization. Preliminary evaluation of fabrics in which selected lead compounds were insolubilized within the cotton showed that, after hot water washing, special properties were imparted to the cotton. Several metals (lead, tin, copper, silver, gold, antimony, and others) have been successfully deposited on and in cotton by a reduction technique applied to certain metal salts. The treated fabrics have good rot and flame resistance, and are capable of being polished to impart a metallic sheen. Cotton fabrics impregnated with lead mercaptobenzothiazole, thiomethyl-, thiopropyl-, and thiophenyltriphenyllead have all exhibited good rot resistance. Some water repellency has been imparted to cotton fabrics by treatment with the lead compounds of N,N'didodecyldithiooxamide and stearyl mercaptan, diethyllead distearate and nickel N,N'didodecyldithiooxamide. Larger scale evaluation of some of the more promising finishes is under way. Attempts are also being made to synthesize new cellulose reactive organolead compounds. (S2 1-202, S2 1-232).

A fundamental study of the preparation and properties of phosphonitrilic and phosphoryl chloride derivatives having potential for reaction with cotton is being conducted under a P.L. 480 grant at Birkbeck College of University of London. The research is an outgrowth of work conducted under P.L. 480 project UR-E29-(20)-35, now expired, under which the chemistry of these interesting inorganic compounds was placed on a sound, systematic basis. Progress is being made in the synthesis, separation, and purification of selected compounds of this type which have configurations that suggest ability to react with cotton cellulose. The information obtained from this work is expected to furnish leads for the development of new reactive finishes for cotton textiles that will confer wash-wear, flame resistance and other desirable properties. (UR-E29-(20)-55).

2. Chemical Reactions Induced in Cotton Cellulose and Chemically Modified Cotton by High-Energy Radiation. In studies of high-energy radiation activated reactions of cotton, the radiochemical yield of graft polymerization reactions of styrene and acrylonitrile onto cotton was found to be influenced by (1) concentration of monomer in treating solution, (2) solvent, (3) ratio of monomer solution to cotton cellulose, (4) prior chemical modification of cellulose, and (5) absence of oxygen, particularly in post-irradiation reactions. The graft polymers, in the form of yarns, were thermoplastic. Yarns grafted with polymethyl methacrylate and polyvinyl acetate by a post-irradiation technique exhibited markedly increased elongation-at-break and decreased average stiffness. Cotton fabrics containing radiation-induced grafted polyacrylonitrile were found to have greatly increased flat abrasion resistance (increases as high as 2000% at 45% add-on of polymer). Radiation-sterilized surgical cotton sutures

(a commercial product) and purified fibrous cotton cellulose were demonstrated to have good post-irradiation stability, an important consideration in such processing.

Although carbohydrates generally degrade on high energy irradiation, experiments conducted on cobalt-60 gamma irradiation of sugars containing aromatic substituents indicated that these groups radiation stabilize the molecules. Work has been initiated to apply these findings to cotton. Preliminary experiments have shown that benzoylation of cotton yarn protects the cellulose from high-energy radiation as indicated by much greater retention of breaking strength in comparison to control yarns. This is the first known example of such protection of a high molecular weight carbohydrate. The discovery could have value in increasing yields of radiation-activated reactions of cellulose with minimum degradation of the fibrous cotton cellulose, and may ultimately prove applicable in the protection of cellulose from degradation by sunlight. (S2 1-176, S2 1-195).

3. Basic Investigation of Reaction Mechanisms, Rates and Catalysis of Cotton Cellulose Reactions. Dimethylol and dialkoxy derivatives of dihydroxyethyleneurea and dihydroxyethylenethiourea were prepared for a fundamental study of mechanisms of their etherification reactions with cellulose. The ring hydroxyl groups as well as the methylol hydroxyl groups of these substituted ureas have been found to react with cellulosic hydroxyls in the presence of inorganic salt catalysts, and the metal ions are complexed with the urea derivatives. The physical and chemical properties of fabrics finished with these agents have been correlated with properties of fabrics finished with equivalent concentrations of dimethylolethyleneurea in the presence of various inorganic salt catalysts at constant metal ion concentration. Data of this type are being accumulated to enable comparison of various etherifying agents as to rates of cellulose etherification reactions and to resultant fabric properties of finished products. Detailed study of the changes in fine structure of cottons finished with butadienediepoxide at room temperature under various conditions of base catalysis has shown that dry crease recovery as well as wet crease recovery can be imparted to cotton in a highly swollen state. (S2 1-196).

A recently initiated line of work is concerned with the preparation of fatty acid or hindered acid esters of cotton, and investigation of the reaction mechanisms involved. Cotton was esterified in nonaqueous media with certain monofunctional acid chlorides to produce cellulosic esters of very low degrees of substitution but with excellent dry and wet crease resistance. This shows conclusively that crosslinking with covalent bonds is not necessarily the only method of obtaining the dry and wet resiliency required in wash-wear cotton fabrics. Requisites of chain size or configuration of the acid chlorides for the imparting of crease resistance will be investigated, as will rates of esterification. (S2 1-233).

P.L. 480 research at the Shirley Institute on the pyrolysis of cotton cellulose conducted under a recently expired grant has provided information

needed for improvement of flame-resistant treatments for cotton. Reduced flammability of cotton textiles for apparel, draperies, awnings, etc., is highly desirable from the standpoint of safety. The nature of the reactions involved in the burning of cotton were studied in three different experimental systems designed to give information on what reactions occur when cotton is heated under various conditions and at different rates, and the sequence of these reactions. It was found that the first products of pyrolysis (oxygen-containing materials such as tars, carbon monoxide, etc.) require little outside oxygen for further combustion and that the degree of flame resistance of cotton fabrics is related to the char to tar ratio upon pyrolysis. This knowledge has shown that fabric finishes designed to reduce the flammability of cotton should be directed toward altering the sequences of these reactions to prevent the formation of combustible products. Certain phosphorus-containing permanent textile finishes were shown to function in this manner. (UR-E29-(20)-9).

4. Exploratory Physical Investigations on Cotton. Completed contract work at the Massachusetts Institute of Technology has shown how and why neps form in cotton. While new neps are formed during textile mechanical processing, the major portion of the increase in the number of neps is due to the breaking down of larger neps initially present in the cotton in the bales as they are received at the mills. The evidence indicates the involvement of mechanical action in every instance of nep formation. Usually, the action is one of rolling of fiber bundles during the processing operation, or the snapback of fibers suddenly released from tension or broken when their tensile yield point has been reached. There is, also, statistical evidence to show a correlation between fiber properties and nep formation. The basic information developed on nep formation will serve as a guide to breeders in developing nonnepping cotton varieties, to machine designers in developing nonnep forming machines and processes, and to machine operators in setting and adjusting their equipment to minimize the formation of neps. (S2 1-173(C)).

The experimental work in fundamental studies to ascertain the effects of chemical modification, fabric construction, and environmental conditions of temperature, relative humidity, and air velocity on the drying rate of chemically modified cotton fabrics has been completed. Processing of computer programs for analysis of the data indicate that vapor diffusion is the controlling mechanism by which water is driven off during drying. Proper drying conditions to minimize resin migration for different crosslinking reactions are predictable. Many interesting features pertaining to the effect of various chemical reagents on the surface morphology and internal structure of cotton cellulose have been determined. The research will make it possible to pinpoint those changes in process drying which are required to minimize drying costs, provide better process control, and improve product quality of various types of chemically modified cotton textiles. (S2 1-188).

Contract research has been initiated by Macrosonics Corporation on the treatment of cotton fibers with acoustic energy and the determination of the effect of such treatments on the physical properties of the fibers. Basic

information of this type is needed for the development of improved equipment for processing cotton into textiles. (S2 1-222(C)).

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AREA NO. 2 - COTTON - INTERRELATIONS AMONG FIBER, YARN, AND FABRIC PROPERTIES

Problem. The intense competition in today's textile markets is placing increasing demands upon cotton producers and processors for high quality products tailored to meet specific use requirements. Improvement in the quality of processed products and lower costs of mechanically processing cotton into yarns and fabrics are needed to satisfy consumer demands and maintain cotton markets. For example, information is needed to determine the effect of the important fiber properties and combination of fiber properties of cottons on yarn and fabric properties and processing performance to obtain the maximum utilization potential from cottons of different fiber properties and to provide guidance for cotton breeders in developing strains having more desirable fiber properties. Improved mechanical processing methods are needed to attain maximum yarn uniformity and the resultant improvements in the general quality level and processing efficiency of all types of cotton products. New and improved methods and instruments for measuring the physical and chemical properties of cotton are needed to guide processing research in developing new and improved products.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving cotton technologists, textile technologists, textile engineers, physicists, statisticians, and mathematicians engaged in research to develop fundamental information and improved processing procedures in order to improve the quality and lower the cost of cotton products during the mechanical processing of cotton fibers into yarns and fabrics.

Research to determine the effect of fiber properties on processing efficiency and product quality is carried out at New Orleans, Louisiana. Additional research of this type is conducted under contract at Auburn Research Foundation, Inc., Auburn, Alabama, involving large-scale spinning evaluations of the effect of fiber properties and spinning variables on yarn properties and end breakage. Cooperation is maintained with cotton merchants and textile mills; the Crops Research Division, ARS, and the Cotton Division, AMS, specially on the procurement of cotton of known history with special fiber properties; and the Market Quality Research Division, ARS, to insure coordination of effort in related research. Research on development of new and improved methods and instruments for measuring the physical and chemical properties of cotton, and evaluating the processing characteristics of cotton, is carried out at New Orleans, Louisiana. Also, contract research is being conducted at Stanford Research Institute, South Pasadena, California, on development of a method for counting neps in cotton at various stages of textile processing.

Other research on effect of fiber properties on processing efficiency and product quality is in progress under grants of P. L. 480 funds to the following foreign institutions: Juan de la Cierva School of Technical Investigations, Barcelona, Spain, for an investigation of the relationship between the cohesion of cotton fibers and the properties of rovings and yarns (project duration - 4 yrs.), and for an investigation of the effect of drafting force on cotton yarn strength and uniformity (project duration - 5 yrs.); Ahmedabad Textile Industry's Research Association, Navrangpura, Ahmedabad, India, for investigation of means to minimize fiber hooked ends in cotton card and drawing slivers (project duration - 4 yrs.). Research on development of new and improved methods and instruments for measuring physical properties of cotton is in progress under a grant of P. L. 480 funds to the following institution: German Research Institute for Textile Industry, Reutlingen-Stuttgart, West Germany, for the development of an apparatus for counting neps in cotton card web (project duration - 4 yrs.).

The Federal in-house scientific effort devoted to research in this area totals 10.3 professional man-years. Of this number 9.5 is devoted to investigations of effect of fiber properties on processing efficiency and product quality and 0.8 to development of new and improved methods and instruments for measuring the physical properties of cotton. The contract research involves an additional 2.2 man-years, 1.0 being on effect of fiber properties on processing efficiency and product quality, and 1.2 on development of new and improved methods and instruments for measuring physical properties of cotton. P. L. 480 research involves 4 grants, of which 3 are on effect of fiber properties on processing efficiency and product quality and 1 on development of new and improved methods and instruments for measuring physical properties of cotton.

The following lines of work were terminated during the year: (1) Basic investigations to characterize fiber damage in mechanical processing from opening through carding to provide information needed to develop improved textile machinery and processing methods, and (2) Fundamental investigation of the causes of warp breakage in weaving of cotton yarns (P. L. 480 project), (under effect of fiber properties on processing efficiency and product quality).

PROGRAM OF STATE EXPERIMENT STATIONS

(A general program statement is given under Area 1)

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Effect of Fiber Properties on Processing Efficiency and Product Quality

1. Effect of Cotton Fiber Properties Such as Length, Strength, Fineness and Elongation on Fabric Properties and Processing Performance. In research to determine the simultaneous effect of pertinent fiber properties and combinations of fiber properties on yarn properties and spinning performance to

provide guides for obtaining maximum utilization of cottons of varying fiber properties, an investigation of the interrelationships of yarn number, yarn twist, and total spinning draft has been completed. The data indicate that yarn strength and elongation at break decreased linearly with increased total draft but decreased curvilinearly with increased yarn number. Also, end breakage in spinning increased curvilinearly with increased draft and yarn number, with the change in yarn number having the greater influence. The relationships found provide the spinning variable bases needed for evaluating the more than 100 samples of cottons, specially selected for their fiber properties, which have been collected for this research. The information should also be useful to mills in improving their spinning performance and product properties through systematic selection of spinning variables. Work is presently in progress to investigate the effect of break drafts, tensor settings, and total draft on end breakage and yarn properties to establish the most suitable combination(s) of draft distribution and tensor settings to use in evaluating the cottons collected. (S2 1-207).

Large-scale spinning evaluations of the effect of fiber properties and spinning variables on yarn properties and end breakage in spinning have been completed by the contractor (Auburn Research Foundation). Commercial spinning tests (25,000 spindle hours) were carried out, on three mixes or blends of cotton (representing two levels each of fiber fineness and fiber strength). Generally, the results of these tests corroborated those obtained with laboratory spinning tests (the SRRL 720 Spindle Hour Test, and the 5000 spindle hour test). Fiber strength and fineness had very little effect on processing performance and quality prior to spinning except in the case of card web neps where the coarser blend produced fewer neps. Fiber strength and fineness had no significant effect upon end breakage during spinning, yarn evenness and appearance, but did have an effect on yarn strength. It was found that fiber length distribution is more important in a blend than either Micronaire reading or fiber strength. Spinning variables such as spindle speed and twist exert a greater influence on end breakage than do fiber properties. Based on the research results, one manufacturer (the cooperator) has completely modified its blending programs, placing greater emphasis on control of fiber length distribution in the blends, and has obtained substantial improvements in processing performance (S2 1-178(C)).

The relationships between the cohesion of cotton fibers and other physical properties of fibers, rovings and yarns are being investigated at the Juan de la Cierva School of Technical Investigations under a P. L. 480 grant now nearing expiration. The cohesion of cotton fibers affects the roll settings, roll pressures and twists to be used in producing yarns of optimum quality. The main laws governing the minimum twist of cohesion of cotton rovings and yarns in connection with testing conditions (length and tension) and fiber parameters (length and micronaire) and yarn parameters (number of fibers per cross section and twist) have been determined; an improved apparatus for measuring minimum twist of cohesion has been developed; and trial work is underway to establish the relationship between fiber surface properties and minimum twist of cohesion. Information developed in this project should permit the relatively rapid and simple measurement of force of cohesion to be

used in predicting the spinning efficiency and yarn properties of cottons of differing fiber properties. (UR-E25-(20)-2).

2. Improved Processing Procedures to Obtain Maximum Utilization of Native and Modified Cottons. The research project to develop optimum processing procedures to minimize the detrimental effect of short fibers on cotton spinning performance and product quality has been terminated. Spinning evaluations of 16 lots of California cottons using the SRRL 720 Spindle Hour Test confirmed previous findings on Arizona cottons that the adverse effects of inferior fiber length distributions on spinning performance can be mitigated by the selection of optimum yarn twists and spinning drafts. Decreasing spinning draft and increasing yarn twist substantially reduces end breakage rates on cottons of inferior length distributions (high short fiber content). The influence of draft is more pronounced on these cottons than on superior cottons. The research findings concerning the relationships between fiber length distributions and spinning variables will enable spinners to select the proper combinations of spinning variables required for optimal spinning performance of cottons having inferior fiber length distributions. (S2 1-179).

Research has been initiated to study principles and procedures for optimum blending of cottons of varying fiber properties. In exploratory processing experiments on two 50/50 mixes of coarse and fine fibered cottons (one with minimum blending; one with maximum) it was found that, while the average finenesses of the fibers in the rovings were equal, the homogeneity (based on variability of fineness along the rovings) of the roving having minimum blending was superior. There were fewer imperfections (as measured by the Neptel instrument) in those yarns processed from the stock having minimum blending. Preliminary indications are that, in processing a blend of fibers differing widely in fineness, preferential feeding may exist and the more machines used in opening and picking, the less the degree of blending obtained. Subsequently, the processing experiments were extended to 50/50 mixes (one with minimum blending; one with maximum blending) of two cottons having wide differences in fiber length distribution (short fiber content), in comparison with similar mixes of a coarse and a fine fibered cotton. Maximum blending produced yarn having skein strength much superior to yarns resulting from minimum blending. Yarn strengths were inversely related to yarn strength variability and to yarn number variability. These results indicate that yarn strength, skein as well as single strand, is a better reflector of insufficient mixing than is fiber bundle tenacity or yarn uniformity as measured by the Uster Evenness Tester. For poorly mixed stock, differences in fineness produced greater differences in processing performance than did differences in fiber length distribution; however, for well mixed stock, processing performance was about equal. (S2 1-234).

The research to characterize fiber damage in mechanical processing of cotton from opening through carding to provide information needed for developing improved textile machinery and processing methods has been terminated. In the carding process, variation in speeds of lickerin, flats, or production caused no significant changes in either fiber lengths or alkali swelling centrifuge (AC) values, but some very slight differences were noted in

spinning performance. Samples from within the running card showed negligible AC value changes after passage through the actual carding area--whether flats or granular surface--but the cylinder-to-cylinder transfers produced measurable increases. Microscopical examination of fibers from card webs after passage through the crush roll attachment of the card showed no unusually damaged areas in the fibers, but fibers which had been subjected to 30 tons per square inch of static pressure in a hydraulic press exhibited excessive damage--bruises, mashing, fractures--extending deeply into the secondary wall. Comparison of important fiber properties of a pair of Deltapine-15 cottons (one harshly ginned and the other normally ginned) after a range of picker-beater actions showed that wide variation in ginning treatment produced no recognizable differences in effects on these properties. These findings are in agreement with results of earlier laboratory studies of fiber damage using the Nepotometer. (S2 1-185).

In the spinning of cotton yarns, assemblies of fibers are simultaneously drawn out and twisted. The drafting forces exerted during the spinning operation affect the quality of the resulting cotton yarns. Research is nearing completion under a P.L. 480 grant soon to expire at the Juan de la Cierva School of Technical Investigations in which an investigation has been made of the effect of various factors in spinning, such as drafting speed, roving twist, apron opening, roll setting, etc., on drafting force in the drafting zones of high draft spinning equipment, and how the drafting forces affect yarn quality. Means have been developed to actually measure the drafting forces in the front and rear drafting zones. Results obtained in the investigations indicate that lowering roving twist and increasing drafting speed increases yarn strength, which means that mills may be able to increase their spinning production with an improvement in yarn strength by this means. The investigation has provided basic information that will be of assistance in developing improved drafting systems, and in making more efficient use of existing systems. (UR-E25-(20)-13).

B. Development of New and Improved Methods and Instruments for Measuring the Physical and Chemical Properties of Cotton

1. Development and Adaptation of Instrumental Techniques for Measuring the Changes Imparted to Cotton by Chemical and Mechanical Processes. Further research was conducted to develop reliable test methods to properly evaluate cotton stretch yarns and fabrics. Three test methods (a static load test, a static extension test, and an extension-cycle test) were evaluated for determining stretch and recovery properties of slack mercerized, resin-treated fabrics. The static load test requires the least time and is also the least complicated of the three. It has been found that by selecting certain conditions of load and time the static load test will give values of growth nearly the same as those obtained with the static extension test. The extension-cycle test will usually rank fabrics according to the amount of growth in the opposite order to that obtained by the other two methods. In testing falsetwisted or backtwisted cotton stretch yarns, the previously developed test procedure for multiple strand specimens has been modified so

that different size yarns can be tested on a comparable basis (a pretension of 0.0006 g./denier and a cycle load of 0.024 g./denier). The test methods developed thus far have proven very useful in evaluating various stretch products from the cotton research program and in guiding research to produce cotton stretch products with improved properties. (S2 1-212).

Neps, small tangled clumps of fibers that first become visible in the card web from the cotton carding machine, are the cause of serious irregularities or defects in cotton fabrics. The counting of neps in the card web is a necessary quality control measure in the production of fabrics, but it is difficult and time consuming when done by the manual methods usually employed. Research under a recently completed P.L. 480 project at the Juan de la Cierva School of Technical Investigations has led to the development of a rapid, automatic scanning device by means of which neps are counted by measurement of irregularities in the transparency of card web samples. This device has been combined with an electronic integrating instrument which will classify the measured neps and irregularities into four groups according to size, and automatically record the number of irregularities in each size group in a given sample of card web. The institution is continuing the development through its own program to find means for distinguishing neps from other irregularities in the card web, thus permitting the counting and classification of neps as distinguished from trash particles. (UR-E25-(20)-1).

P.L. 480 research is continuing at the German Research Institute for Textile Industry to develop an apparatus for the rapid and automatic counting of neps in cotton card web by means of light reflectance and detection. Further progress has been made toward the development of an instrument capable of automatically and continuously measuring and recording neps in the web from a full scale card, based on a prototype developed for use with a laboratory scale card. The development, if successful, is expected to be of great value to cotton processors since it would provide rapid means for following, and perhaps automatically controlling, an important processing variable that affects cotton fabric quality. (UR-E10-(20)-2).

PUBLICATIONS -- USDA AND COOPERATIVE PROGRAMS

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Development of New and Improved Methods and Instruments for Measuring the Physical and Chemical Properties of Cotton

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1/ Publication resulting from research under grant of P.L. 480 funds to the foreign institution.

AREA NO. 3 - COTTON - NEW AND IMPROVED TEXTILE MACHINERY

Problem. Cotton is plagued by problems of trash and nonuniformity of fiber length distribution that are not present in synthetic fibers, paper, and other competitive products. Highly efficient methods of cleaning are needed by the cotton textile industry to process satisfactorily the large quantities of machine-harvested and roughly hand-harvested cottons being marketed. Last year 71% of the cotton crop was harvested by these methods in the United States. Such cottons are difficult to clean with existing textile equipment because of the type of their trash -- largely fine, leaf trash. The development of an integrated system for opening, cleaning, and carding today's cotton can provide substantial improvements in quality and lower costs. The present cotton mill utilizes ten or more processing stages and, compared with other manufacturing systems, an excessive amount of labor. The redesign of existing equipment and the development of radically new types of processing machinery offers an opportunity for major improvements in uniformity and overall quality of textile products, and for savings in manufacturing costs through decreased waste of spinnable fiber, and through reduction in machinery investment, space, and labor.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving mechanical engineers, physicists, and cotton technologists engaged in research to design and develop new and improved equipment for processing cotton into higher quality, lower cost consumer products.

Research to develop improved mechanical processing machinery, for opening through carding, is conducted at New Orleans, Louisiana. This work includes the development of experimental machines and pilot scale machines for evaluation under pilot-plant conditions, and subsequent development of plans for scaling up successful units into practical, commercial size equipment. Current research involves the development of a bale-breaker blender for opening and blending cotton, the improvement of cleaning at the card, and the development of a machine for removing short fibers from cotton. Close cooperation is maintained with cotton textile machine manufacturers and cotton textile processors in the establishment and dissemination of engineering specifications for the commercialization of new and modified processing equipment. Additional research in this area is being conducted under contract at General Applied Science Laboratories, Inc., Westbury, L. I., N. Y., on the aerodynamic separation of lint cotton into individualized fibers to provide information needed for improving cotton textile processing equipment.

The Federal in-house scientific effort devoted to research in this area totals 16.4 professional man-years. All of this effort is on the development

of improved mechanical processing machinery - opening through carding. The contract research involves an additional 1.1 man-years, in the field of improved mechanical processing machinery-opening through carding.

PROGRAM OF STATE EXPERIMENT STATIONS

(A general program statement is given under Area 1)

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Improved Mechanical Processing Machinery - Opening Through Carding

1. Equipment for Blending Cottons of Different Fiber Properties to Produce Improved Cotton Textiles. Further research on the pilot model, of half-size, bale-breaker blender for opening and blending cotton has culminated in the development of a prototype machine capable of opening and blending a "sandwich" or composite bale consisting of as many as fifteen or more layers from different bales of cotton, at an equivalent production rate of a bale an hour in a full size machine. It has not proven possible to operate the pilot machine efficiently at production rates much higher than this. A mechanical sandwich-bale compression system incorporated into the prototype machine to enable processing more cotton per loading was found to be unsatisfactory from an operational and economic standpoint and has been abandoned.

Work is now being initiated under a new project to scale-up and modify the design of the pilot model blender into a full size machine, and to evaluate this machine under laboratory and mill conditions. Several modifications of the pilot blender will be tested prior to undertaking final design of the new full size machine. (S2 1-154, S2 1-252).

2. Improved Cotton Carding Machinery for Better Cleaning, Fiber Separation and Orientation. Research directed toward the improvement of cleaning at the cotton card has continued. An improved method of feeding the card is needed to overcome existing limitations in trash removal and fiber separation. A Precarding Feed System has been designed, constructed, and installed. All mechanisms are operating satisfactorily. The doffer-transfer cylinder appears to perform best at relatively high speed. The precarding mechanism is undergoing evaluation as an individual machine, i.e., separate from the card, to enable evaluation of the quality of web and sliver as a sensitive indicator of effects achieved by changes made in the mechanism.

Additional investigations of a conventional card feed system have shown that its efficiency is independent of speed and production rate when the relative motion of all units is maintained. There was no loss in cleaning and web quality with speeds and output increased to over twice normal. Basic studies established that when the normal positive air pressure (about 1/16 inch water) existing above the feed point of the card is lowered to zero by an external source of suction, the efficiency of the machine is improved.

Trash removal is increased and loss of spinnable fibers is decreased. Lowering the pressure into the negative range of 0 to -1 inch water is detrimental.

Another phase of work involves development of a suitable mechanism for drafting picker laps for feeding the card. An experimental lap-drafting apparatus utilizing conventional drafting rolls proved unsuitable. Therefore, a completely new type drafting apparatus was designed and a bench model constructed. Design and operational variables are presently under investigation. Initial results indicate that the new approach has promise for improving the quality of products from the cotton card, and the quality of cotton yarns. (S2 1-215).

3. Machine for Removing Short Fibers from Cotton. Research was continued on the application of uniform and nonuniform electrostatic field fractionating devices to the problem of removing short fibers from cotton. Means for feeding individualized fibers to the devices at higher rates have been developed to increase production. The increased quantities of individualized fibers can now be supplied to a point where degradation in the quality of electrostatic fractionation occurs, due largely to the interference of air currents generated by the feed system. Numerous modifications of the fractionating devices have been investigated in attempts to increase production rates to a reasonable level while maintaining high percentage short fiber removal. In all instances increasing the production resulted in a rapid decrease in fiber fractionation. A high speed photographic study is being conducted to determine if a relationship exists between fiber length and electrostatic attraction. Such a relationship will be helpful in designing more efficient electrostatic fractionating devices. Use of mechanical forces to fractionate fibers is also under study. An apparatus combining aerodynamics with mechanics was designed, constructed, and will now be evaluated. (S2 1-164(Rev.)).

4. Aerodynamic System for Separating Lint Cotton into Individualized Fibers. An analytical investigation of the application of aerodynamic forces for individualizing cotton fibers is being conducted in contract research at General Applied Science Laboratories, Inc. The contractor has made a theoretical study of the flow of fibers in a viscous fluid (air) as the first phase of the investigation. A system of equations has been derived for predicting particle motion in air. In this study, the particle was assumed to be an elongated ellipsoid of revolution to approximate the geometry of a cotton fiber. The results of this analysis provide information on the aerodynamic force characteristics of the fiber and are to be used in the study of fiber motion in nonuniform flows. A series of experiments was also conducted to study whether substantial fiber separation could be accomplished by rapidly accelerating a tuft through an orifice. Data obtained with existing equipment over a range of pressures will be utilized in designing a new apparatus for achieving improved fiber separation. If a satisfactory system for individualizing cotton fibers can be devised, this would enable the scientific design of a single machine for opening, and

possibly cleaning, lint cotton to process cotton more efficiently into higher quality textiles at lower cost. (S2 1-204(C)).

PUBLICATIONS -- USDA AND COOPERATIVE PROGRAMS

Improved Mechanical Processing Machinery -- Opening through Carding

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Improved Mechanical Processing Machinery - Drawing Through Weaving

Kyame, George J. and Copeland, Herbert R. 1964. Ringless spinning updated. Textile World 114(3), pp. 48-51.

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AREA NO. 4 - COTTON - IMPROVEMENT OF WASH-WEAR PROPERTIES

Problem. Garments which are wrinkle resistant and suitable for wash-wear use are increasingly important to the consumer. Although much progress has been made toward securing this market for cotton, much additional information is needed to hold and expand cotton's share of this enormous market. According to current industry estimates 1.2 million bales of cotton are used annually which would not have been utilized except for the wash-wear development. Projected estimates indicate that in the future most apparel and almost all household textiles will be given a wash-wear or a minimum-care finish. Research on synthetic fabrics is mainly aimed at this lucrative market and is several times greater than the entire utilization effort on cotton. At the same time chemical firms are reducing their research in the development of cotton wash-wear finishes. Promotional advertising claims on cotton wash-wear products have exceeded the actual achievement, and many problems remain to be solved. Much fundamental information is needed to explain mechanisms of the reaction of cotton with crosslinking agents as a basis for the development of new and better wash-wear finishes and for the improvement of present processing techniques. Much applied information is needed which, while essential to the maximum utilization of cotton, is generally beneficial to all processors and therefore comparatively unattractive financially to individual companies. Areas in which research is needed to improve wash-wear cottons include processing techniques, fabric appearance, durability, and comfort. Fabric appearance involves the ability to dry smoothly, resistance to wrinkling or mussing during wear, resistance to dry, wet, and oil soiling, introduction of durable creases as desired, dimensional stability and elimination of seam pucker. Durability involves tensile and tearing strength and abrasion resistance in the finished fabric as well as resistance to abusive laundering, particularly bleaching and souring. Comfort involves moisture absorption during use, elimination of odor on storage or wearing and, in certain cases, stretchability of fabric.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving organic chemists, physical chemists, analytical chemists, physicists, microscopists, chemical engineers, mathematicians, cotton technologists, textile technologists and textile engineers engaged in both basic and applied research on wash-wear finishing and improvement of wash-wear properties of cotton. Informal cooperation is maintained with textile finishers, chemical manufacturers, and textile research institutes in connection with the research.

Basic and exploratory research on wash-wear finishing of cotton is conducted at New Orleans, Louisiana. This research is designed to give a better understanding of the chemical reactions and physical changes taking place in wash-wear finishing and the crosslinking of cotton in general. It also

seeks to correlate the properties of the finished cotton with the nature of the crosslinking agent. Basic studies of the relationship of fiber properties to fabric behavior in wash-wear treatments are also conducted. The results provide a broad and sound foundation for the development of new practical wash-wear finishes for cotton. Research on the improvement of smooth drying properties -- the essential features of a wash-wear fabric -- is conducted at New Orleans, Louisiana. Some important phases of current work involve development of new crosslinking treatments and optimum wash-wear fabric structures; combination of chemical and mechanical treatments to improve strength and resilience; and pilot-plant evaluation of promising laboratory finishes. Additional research on improved smooth drying properties is in progress under contract at the Fabric Research Laboratories, Dedham, Massachusetts, on investigation of the relationships between fabric structure and ease-of-care performance; and at North Carolina State College, Raleigh, North Carolina, on the effects of mechanical treatments of fabrics prior to, during and following resin finishing on ease-of-care properties.

Research to develop new and improved processing methods for the production of wash-wear cotton yard goods and garments is carried out at New Orleans, Louisiana. Processing methods are being investigated for the production of wash-wear cotton stretch fabrics with improved strength, drape and hand. Methods of crosslinking stretch cotton to stabilize the fabric and make the stretch durable to laundering are undergoing study. Cost estimates for new chemicals and for processing of cotton are made to aid industrial establishment of the research developments. Additional processing research is being conducted under contract at Georgia Tech Research Institute, Atlanta, Georgia, to develop improved cotton sewing thread for wash-wear fabric structures, compatible with existing high-speed manufacturing methods, which will not cause seam pucker, or which will have a markedly reduced tendency to cause seam pucker.

The Federal in-house scientific effort devoted to research in this area totals 29.1 professional man-years. Of this number 8.4 is devoted to basic and exploratory research on wash-wear, 15.4 to research on improved smooth drying properties, and 5.3 to new and improved processing methods. The contract research involves an additional 2.9 man-years, 1.7 being on improved smooth drying properties, and 1.2 on new and improved processing methods.

PROGRAM OF STATE EXPERIMENT STATIONS

(A general program statement is given under Area 1)

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Basic and Exploratory Research on Wash-Wear

1. Basic Studies of Recovery from Wrinkling and Creasing. Several lines of investigation of wet and dry crease recovery mechanisms in wash-wear cotton

products have continued. In physical behavior studies, the immediate tensile recovery (first few seconds) has been found to be the most informative tensile measure to differentiate wash-wear treatments and mechanisms of recovery. It is the most sensitive to test conditions and modifications and the most improved when achieving wash-wear properties. The tensile recoveries of mercerized, formaldehyde-treated cotton yarns indicate that slack mercerization decreases permanent set and increases delayed recovery. The increase in delayed recovery, greater under wet than under standard conditions, is achieved by decreases in immediate recovery. In extending the work to fabrics (cotton printcloth treated by several formaldehyde crosslinking methods), it was found that recovery differences due to testing humidity and treatment are twice as large when measured on the yarns as on fabrics. The tensile recoveries have shown differences between wash-wear treatments which could not be observed from crease recovery and could offer explanation for differences between crease recovery and performance in many fabrics. Density values of the cottons were found to decrease with increasing chemical crosslinking and with increasing water content of the formaldehyde treating solutions.

Electron microscopy investigations have shown that structural members of the cotton fiber can be immobilized by crosslinking and the fiber rendered unresponsive to cellulose swelling and dispersing agents. Application of a technique, developed for separation of the cell wall lamellae in the cotton fiber, demonstrated that inter-lamellar and intra-lamellar crosslinking are correlated with the chemical history and physical behavior of the fiber. Inter-lamellar crosslinking is usually associated with high dry-crease recovery; less inter-lamellar bonding in crosslinked cotton usually corresponds to higher wet- and lower dry-crease recovery.

In other work it was established that the use of additives to preserve swelling in dry-cure crosslinking raises moisture regain but not water-of-imbibition, while wet crosslinking with increasing swelling raises both. This indicates that there are at least two separate mechanisms involved in the general term swelling, and points to the possibility that desirable properties of cotton can be increased without undesirable side effects by use of specific changes in absorption swelling. The use of additives in dry-cure crosslinking processes has practical possibilities. (S2 1-189; S2 1-262, Pending).

Recent research on relationships of fiber properties to fabric behavior in wash-wear treatments has involved investigations of the correlation of wrinkle recovery and elastic recovery for various types and structures of wash-wear fabrics. A higher correlation of wrinkle recovery to elastic recovery was found for printcloth than for basket weave, and for high twist than low twist in yarns, when elastic recovery was measured at 4% strain. When fabrics are grouped according to their modulus into low, medium, and high, the correlations improve more for the basket weave than the print. Since the printcloths are the more rigid fabrics, the effects of rigidity possibly affect the relationships. The previously reported inverse

relationship between resin add-on and density for crosslinked cottons was found not to be affected by yarn twist but it is affected by fabric weave. A loose weave fabric is reduced more in density, at a given add-on, than a tight weave fabric. Tension during resin treatment reduces density in the case of prescoured fabrics, but increases it in the case of slack premercerized fabrics. The inverse relationship between density of resin treated fabrics and Monsanto Wrinkle Recovery is affected by cloth type, pretreatment, extent of resin treatment, and tension applied during treatment. Progress in understanding the complex relationships in translation of fiber properties to such fabric properties as wrinkle recovery, bending rigidity and strength in fabrics of different structures, and the effects of the variables in resin treatments on fiber and fabric properties, will aid attempts to alter fiber properties by chemical and physical methods to achieve improved wash-wear fabric properties. (S2 1-198).

Initial findings concerning the effects of time and environmental conditions on the rate of wrinkle recovery of wash-wear cotton textiles were confirmed when the studies were extended to numerous types of crosslinking agents. Differences in rate of recovery due to finishing agent used are most pronounced in the first 10 seconds of the test. Recoveries after 10 seconds, and especially after one minute, appear to be an effect of the level of treatment instead of the type of finishing agent. It appears that for the highest level of wrinkle resistance, agents imparting high immediate crease recovery are most desirable. Tests conducted at different relative humidities showed that the recovery angles at 45% and 65% R.H. are generally higher than at 30% R.H. APO finished cotton seems to be unique in that it shows a maximum recovery angle when creased at about 90% R.H. and allowed to recover at 65% R.H. In contrast, cotton finished with dimethylol ethyl triazone or urea formaldehyde appears to exhibit this maximum when creased and recovered at 65% relative humidity. Wet crease recovery angles of fabrics finished with these three crosslinking agents are lower than those determined under the atmosphere of the standard test conditions. As moisture content of wetted APO finished cotton is increased, the recovery angle is decreased. Since crease recovery properties of wrinkle resistant cottons depend upon atmospheric humidity and moisture content of the fabric, and these recovery properties are different for various finishes, it is possible that a specific finish may be more suitable for use in a particular climate or geographical area. (S2 1-203).

B. Improved Smooth Drying Properties

1. Development of Treatments to Improve Strength, Resilience and Other Desirable Properties of Wash-Wear Cottons. Research has been initiated under a new project to produce wash-wear cotton fabric with improved smooth drying properties, wet crease recovery, and moisture absorptivity by using treatments that swell the cotton before or during crosslinking. Inert inorganic salts have been used in dry-cure crosslinking to improve moisture regain and swellability in the treated cotton fabric; these salts can also increase reactivity in wet crosslinking treatments. The inorganic additives

are less expensive and more attractive for commercial use than the organic additives previously reported, and will be investigated further. Swelling treatments such as mercerization give greater swellability in crosslinked cotton if water washing and evaporation of water are avoided. Swelling by acids and salts is ineffective if water washes are used. (S2 1-235).

The production of excellent smooth drying and wrinkle resistant fabrics by crosslinking cotton with highly reactive methylolamide amino acid derivatives is being sought in recently initiated research. Four poly 2-carbamolylethyl derivatives of amino acids (glycine, alpha alanine, beta alanine and lysine) have been prepared, methylolated, and used to produce wash-wear cottons. These agents have proven to be highly reactive to cotton but, in general, are slightly susceptible to chlorine damage. Major effort has been on the glycine and alpha alanine derivatives, which can be isolated and purified. The lysine agent has been blended with some success with urea-formaldehyde and formamide-formaldehyde adducts. Synthesis of similar agents with phosphorus acid groups has been unsuccessful thus far.

Several formaldehyde-free, N-methylol crosslinking agents have also been prepared. Succinimide, N-methyl formamide, and 2-pyrolidone add to glyoxal to form compounds with no NH groups. N,N'-dihydroxyethylene-bis(2-pyrolidone) offers the most promise of these agents. Use of formaldehyde-free agents of this type could eliminate odor problems and also reduce costs of wash-wear finishing by elimination of the afterwash. (S2 1-227).

Research is in progress to determine optimum conditions for the application to cotton of dimethylol monoalkyl carbamates to produce durable wash-wear finishes. The effects of changing the reaction conditions--time, temperature, reagent ratios, pH, nature of the alkyl substituent, presence of N-substituents--in the methylolation of monoalkyl carbamates have been established. This fundamental information should be of much practical value by providing the best routes to the production of the carbamate crosslinking agents. Finishes from three of the methylolated products have exhibited outstanding durability to repeated commercial laundering under rigorous conditions of hot alkaline washing, chlorine bleaching, and acid souring. A citric acid-magnesium chloride mixed catalyst system has proven effective for carbamate finishing at relatively low (120° C.) curing temperatures, as well as at conventional curing temperatures (160° C.) for very short times (30 sec.). Preliminary toxicity tests conducted by the Pharmacology Laboratory, WU, using rabbits as the test animal, indicated no evidence of skin irritation from carbamate-finished cotton. Interest by industry in carbamate finishes continues to grow. Several companies are now offering dimethylol alkyl carbamate finishing agents on a commercial basis. (S2 1-230).

Contract research at North Carolina State College to determine the effects of mechanical treatment of cotton fabrics prior to, during, and following resin finishing on the ease-of-care properties of the fabrics is progressing satisfactorily. The effectiveness of DMEU resin on wash-wear rating was found to be reduced by 10 commercial launderings but not equally for fabrics

subjected to the different stretching and compressive shrinkage processes. The reliability of these observed differences will be more accurately assessed after other type crosslinking treatments are evaluated. Physical tests have been completed on all APO resin-treated fabrics, and statistical evaluation of the data in relation to treatment variables is currently in progress. Progress is also being made in treating the various fabrics with the ethyl carbamate finishing agent. (S2 1-183(C)).

2. Development of Optimum Wash-Wear Fabric Structures. The research project to develop optimal structures for cotton fabrics for wash-wear products has been terminated. The research has confirmed the findings of others that fabric structure plays no important part in improving the smooth drying properties of a fabric, but does improve such physical properties as strength (tear in particular) and abrasion resistance. However, improvements due to structure in the basic, unfinished fabric are in a large measure nullified on finishing and crosslinking. Two methods for providing "built-in" stress relief within the fabric structure (via additional yarn length between cross-yarn contacts) were investigated: (1) a chemical method (slack mercerizing an open weave fabric), and (2) a physical method (weaving in the additional yarn length at the loom). The "built-in" stress relief approach failed to give the improvement in wash-wear properties expected on a theoretical basis. This may have been due to the inability to crosslink and cure the test fabrics in a smooth state under tensionless conditions. (S2 1-163).

Analyses of data obtained in the contract research at Fabric Research Laboratories on relationships between ease-of-care performance and geometry (structure) of cotton fabrics have been completed. The research has shown that wrinkle recovery measurements can give a reasonable guide to wash-wear behavior. They are less applicable when marked changes in fabric stiffness occur. If flexural rigidity is increased, the wash-wear rating for any given level of wrinkle recovery is apt to increase. The testing atmospheric condition which showed the best relation between wrinkle recovery and wash-wear behavior was 120° F., 15% relative humidity. Ease-of-care characteristics achievable by structure differences were found to be small in the case of fabrics not tightly woven. In dense fabrics, advantages are to be gained from weaves of long floats to increase yarn mobility. In both open and closed weave fabrics, increases in yarn mobility resulted in increased tearing strength. Mercerization increased both tearing strength and wrinkle recovery characteristics. (S2 1-170(C)).

C. New and Improved Processing Methods

1. Wash-Wear Cotton Stretch Fabrics With Improved Strength, Drape and Hand. Good progress has been made in research investigations of finishing treatments to produce wash-wear cotton stretch fabrics with improved strength, drape and hand. Restretching slack mercerized cotton fabrics to original dimensions, then crosslinking, has yielded fabrics with much greater breaking and tearing strength than the crosslinked control fabrics. The effect that yarn and fabric structure have on the improved strength achieved by the

restretching process is being investigated. The most significant changes due to yarn structure and mercerization have occurred in the filling properties. For textile structures studied to date, twist multiplier had little effect on warp breaking strength properties whether the fabric was or was not mercerized before crosslinking.

It has been found that drapability of slack mercerized printcloth is slightly improved by use of polyethylene softener, but the improvements due to crosslinking with gaseous formaldehyde are greater. Variables in gaseous treatments for producing improved wash-wear and strength properties are being determined. Dimethylol ethyl carbamate treated fabric, cured with gaseous catalyst, has good wrinkle resistance, strength, and resistance to chlorine damage.

The discovery that swelling of the cotton fibers can be increased by diluting the caustic in the impregnated fibers after the mercerization treatment will be investigated from the standpoint of establishing optimum conditions for restretching to improve strength properties of the finished fabric. Wet crease recovery improvement of fabric has been achieved by carboxymethylation in conjunction with slack mercerization; modifications of this technique will also be studied. (S2 1-211).

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AREA NO. 5 - COTTON PRODUCTS WITH SPECIAL PROPERTIES

Problem. In many uses where special properties are of paramount importance, cotton is being replaced by synthetic materials. To improve its position in the textile market, which has declined from 79.5% of mill consumption of all textile fibers in 1939 to an estimated 56% in 1963, new applications must be explored and improved products developed to meet the competition of synthetic fibers. Cottons having high recoverable stretch, durable loft, light-weight bulk, pleasing textures, warmth and other highly desirable properties are needed to enable cotton to compete successfully with synthetic fibers in the rapidly expanding market for stretch and bulked type fabrics. Fabrics designed to achieve increased resistance to tearing and abrasion, flex life and other strength properties are needed to improve the wear life of cotton textiles for apparel, household and industrial uses. Cotton fabrics must be designed to withstand better the elements of weather and finishes developed that will provide greater protection from solar radiation, microorganisms, acids and fire, and that will resist color change. Additional basic information must be developed to improve cotton's resistance to water and oil-borne soils, and to dry soiling. Resistance to soiling ranks fifth in importance among the 40 end-use qualities for textiles. Cheaper and durable flame retardant finishes for cotton, specially for outdoor use, are needed. Numerous consumer preference surveys have shown that a great potential demand exists for cotton material that will be more lustrous without sacrifice of functional properties. Cotton textiles with multipurpose finishes are also needed, particularly those where several desirable end-use properties can be introduced in a single process.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving organic chemists, analytical chemists, physical chemists, physicists, microscopists, chemical engineers, cotton technologists, textile technologists and textile engineers engaged in both basic and applied research to develop new or improved cotton products possessing special properties to meet the competition of synthetic fibers and other synthetic materials in various end uses. Informal cooperation is maintained with textile finishers, chemical manufacturers, and textile research institutes in connection with the research.

Research is carried out at New Orleans, Louisiana, in cooperation with the Foundation for Cotton Research and Education (affiliated with the National Cotton Council of America) and the Canvas Products Association International, to develop cotton fabrics with improved resistance to outdoor weathering. This research includes discovery of new and more effective biocides, and sunlight-resistant pigments for cotton textiles; and development of improved formulations, equipment and procedures for producing weather resistant cotton textiles. Additional research is being conducted under contract

at Texas Woman's University, Denton, Texas, on development of weather-resistant, water-repellent finishes for cotton; and at Southern Research Institute, Birmingham, Alabama, on investigation of interfacial and graft polymerization procedures for producing weather-resistant cotton textiles with improved physical properties.

Research to develop new fluorochemical finishes for oil- and water-repellency and other reactive and additive finishes is conducted at New Orleans, Louisiana, to improve cotton's soil resistance. Additional research is being performed: (1) under contract at the Harris Research Laboratories, Inc., Washington, D. C., to provide fundamental information on the mechanism of the soiling of cotton by dry soils, and water-, oil- and solvent-borne soils, which could lead to the formulation of a general theory of the soiling of cotton and modified cotton; and (2) under a grant at the University of Arizona, Tucson, Arizona, on correlation of surface microtopography of treated and untreated cotton fibers with resistance to soiling of cotton textiles.

Research on flame resistant cotton textiles is performed at New Orleans, Louisiana. Recent emphasis has been on the development of treatments to impart flame resistance to cotton while at the same time imparting other desired textile properties.

Investigations of methods for imparting durable luster and related appearance characteristics to cotton textiles are carried out at New Orleans, Louisiana.

Research to improve cotton's bulk, elasticity and resilience through resin treatment, chemical modification, slack mercerization and other type swelling treatments, of fibers, yarns and fabrics is conducted at New Orleans, Louisiana. The research on fibers is aimed primarily at the development, by chemical or mechanical means or both, of more resilient and cohesive cotton batts for use in mattresses and other padding applications in the furniture and automobile industries. The cotton batting research is conducted cooperatively with the National Cotton Batting Institute, Textile Waste Association, National Cottonseed Products Association and the Foundation for Cotton Research and Education (affiliated with the National Cotton Council of America). Work on yarns is intended to produce bulky, elastic yarns suitable for weaving or knitting into fabrics with improved stretch and bulk characteristics. Investigation of a slack mercerization process, with and without subsequent resin treatment, is being carried out to achieve improved stretch cotton fabrics for industrial, household and apparel uses. Additional research on stretch and bulked cotton products is being carried out under contracts at North Carolina State College, Raleigh, N. C., on evaluation of stretch-type cotton yarns (prepared by backtwisting and false-twisting techniques) in knit wear; and on determination of optimum yarn constructions, knitting structures and prefabrication design for producing stretchable articles of knitted cotton wearing apparel by slack mercerization; and at Clemson Agricultural College, Clemson, S. C., on development

of cotton knit fabrics having increased bulk, warmth, and dimensional stability by application of finishing agents.

Research on the effect of yarn and fabric construction on the physical properties of chemically treated cotton fabrics is another phase of work at New Orleans, Louisiana. Research is in progress to determine the influence of yarn and fabric structures on the properties of stretch cotton fabrics produced by slack mercerization, with and without subsequent resin treatment.

The Federal in-house scientific effort devoted to research in this area totals 32.8 professional man-years. Of this total, 5.4 is devoted to weather resistant cotton fabrics, 1.0 to soil resistant cotton textiles, 6.6 to flame resistant cotton textiles, 2.5 to cotton textiles with improved luster, 14.6 to stretch and bulked cotton products, and 2.7 to effect of yarn and fabric construction on the physical properties of chemically treated cotton fabrics. The domestic contract and grant research involves an additional 6.9 man-years, 1.5 being on weather resistant cotton fabrics, 2.3 on soil resistant cotton textiles, and 3.1 on stretch and bulked cotton products.

The following lines of work were terminated during the year: (1) Design and development of acceptable cotton crepe apparel fabrics to compete with synthetic fibers in these markets, and (2) An engineering study of the feasibility and practicality of chemical and/or resin treatment of roving by continuous processing as an intermediate step in the mechanical processing of cotton (under effect of yarn and fabric construction on the physical properties of chemically treated cotton fabrics).

PROGRAM OF STATE EXPERIMENT STATIONS

(A general program statement is given under Area 1)

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Weather Resistant Cotton Fabrics

1. Improved Biocides, and Sunlight-Resistant Pigments for Cotton Textiles; and Improved Formulations, Equipment, and Procedures for Producing Outdoor Cotton Textiles. Cotton fabrics treated with the zirconyl ammonium carbonate - copper borate fungicides developed in cooperative research with the Canvas Products Association International and the Foundation for Cotton Research and Education have shown high resistance to rot in soil burial tests. The fabrics retained 100% of their strength after 5 weeks' burial. After 12 months of outdoor weathering, there is no evidence of growth of mildew and algae on the fabrics. Cotton duck treated with the copper zirconyl boroacetate finish has shown no evidence of microbiological attack and a high strength retention after fourteen months of outdoor exposure. Several companies interested in the treatment of outdoor cotton fabrics are already looking into the possibilities of these new finishes. Mineral dyeings of

cotton fabrics have been effected with certain chromium and nickel compounds solubilized and then redeposited with zirconium salts. These agents can be employed together with the copper borate - zirconium salt fungicides to impart light screening and antimicrobiological activity in a single treatment. The substantial areas of promising new research discovered will be investigated under new projects. (S2 1-156).

Experimental work has been initiated in contract research at Texas Woman's University to develop weather-resistant, water-repellent finishes for cotton. Following completion of a survey of commercial water-repellents, and the major part of a literature search covering the area of cotton water repellency, the contractor has applied a number of selected water-repellents to cotton duck and twill and placed the treated samples on exposure. These agents are from a list of approximately fifty water-repellents, including at least two from the twelve general types, compiled by the contractor. The application of finishes and their evaluation will continue. (S2 1-200(C)).

B. Soil Resistant Cotton Textiles

1. Fluorochemical and Other Soil Resistant Finishes for Cotton. Modification of cotton with fluorochemicals to impart durable water and oil repellency has proceeded along two lines--chemical reaction, and a combination of reaction and polymerization. Only the latter approach has yielded useful results to date. Ethylenimine has been found to be a useful material for linking the cellulose chain to long chain fluoroacyl compounds such as ethyl perfluorooctanoate. The latter treatment imparts durable oil repellency to fabric at low add-ons, and fair water repellency. Some progress has been made in developing methods of synthesizing alpha-chloroalkyl perfluoroalkyl ethers; however, due to instability of these ethers it has not been possible to obtain products of the desired purity for treatment of cotton. In other work, simple procedures have been devised for preparing primary and secondary perfluoroamines by sodium borohydride reduction of the corresponding perfluoronitriles and perfluoroamides, respectively. Although the amines themselves are not cellulose-reactive, they are capable of reaction with compounds such as THPC which are reactive with both cotton and the fluorocarbon. In certain cases this combination has given water and oil repellency although the treated fabric is colored and has poor tensile strength. Further work is in progress using this approach. Also, additional research will be conducted to improve the ethyl perfluorooctanoate-ethylenimine oil- and water-repellent finish. (S2 1-180, S2 1-250).

The in-house investigations of various finishes with respect to soiling and soil removal from cotton have been terminated. Further work showed that cotton fabrics with hydrophobic finishes or cationic surfaces require higher detergent concentrations in the wash to remove soil. Soil is readily removed from carboxymethylcellulose (CMC)-treated and phosphonomethylated (anionic) fabrics at low as well as high detergent concentrations. It was established that CMC can be added to crosslinking formulations based on triazines and ethyleneureas to produce wash-wear cotton fabrics with improved resistance

to aqueous and oily soils. The use of the CMC also makes soil easier to remove by laundering techniques. This improvement was durable through 25 launderings. A resin-CMC add-on of approximately 3-4% applied from a solution containing approximately 3-5% resin forming monomer and 2% medium viscosity CMC produced the best finish with respect to soil resistance and ease of soil removal. No problems were encountered in scaling up the application of these finishes to cotton fabric as evidenced by two successful pilot-plant runs. Industry has shown interest in this soil resistant finish. (S2 1-191).

Fundamental studies of soiling and soil removal in aqueous and oily systems, under a contract project at Harris Research Laboratories, Inc., have been completed. A comparison of the results of streaming potential and negatively charged soil (ferric oxide pigment) deposition measurements on cotton fabrics with various finishes showed that even relatively large changes in the degree of negative zeta potential of the fabric do not produce the expected corresponding change in deposition of the nagatively charged pigment. In another phase of work, the surface energy of cotton finishing materials as characterized by wetting measurements was determined to ascertain the relation between surface energy of the finishes and their tendency to become soiled and the ease with which soil is removed. It was found that the capillary structure of cotton fabrics substantially raises the critical surface tension for wetting above the value for smooth surfaces. The wettability by organic liquids of fabrics immersed in water is inversely related to their wettability in air. Silicone, fluorocarbon, and acrylic polymer finished fabrics immersed in water have strong affinities for organic liquids compared with untreated cotton. The deposition from water of hydrophobic solid soil onto cotton finishes increases with increasing oil wettability of the finish immersed in water. Hydrophilic pigment deposition does not depend upon the degree of oil wettability of the finish. Laundering at high temperatures causes the more polar oils to become firmly fixed to fabrics. In general, organic finishing agents more hydrophobic than cellulose make oily soil removal more difficult than from untreated cotton. (S2 1-175(C)).

Based on results from the completed research, new contract research has been initiated at Harris Research Laboratories to determine if a high energy interface between the surface of the fiber and its environment leads to heavy soiling in general. The effect of soiling environment on the soiling tendency of a series of selected cotton finishes will be determined and an attempt will be made to formulate a general theory of the soiling of cotton and modified cottons. The Southern Division has supplied base fabrics for initial phases of the soiling studies. A significant step has already been made by devising what appears to be a suitable dry soiling procedure for cotton. This is very important since no suitable method exists in which cotton can be dry soiled at a uniform level. (S2 1-223(C)).

C. Flame Resistant Cotton Textiles

1. Treatments to Impart Flame Resistance and Improved Textile Properties to Cotton. Research to develop improved treatments for imparting flame resistance and other desired textile properties to cotton has continued. Three new flameproofing formulations were prepared which impart good flame resistance to cotton at relatively low weight gains. Precondensates of THPC with tris(2-carbamoylethyl) phosphine (TCEP), tris(2-carbamoylethyl) phosphine oxide (TCPO), or phosphoroxytriamide (PTA) are employed and fixed on the cotton with ammonia. Cotton fabric treated with the THPC-PTA precondensate proved particularly interesting in that it showed essentially no strength loss after chlorine bleaching and scorching. Moderate flame- and rot-resistance and excellent crease resistance were simultaneously imparted to cotton fabrics by application of the methylol derivatives of TCEP and TCPO. The finishes are durable to repeated home launderings. In recent work it was discovered that good flame retardancy can be imparted to cotton textiles by fixing THPC alone in the textiles with ammonia. This process is simpler and cheaper than the earlier ones employing precondensates of THPC, and the finish is more resistant to removal by laundering. The process is undergoing evaluation in industry.

A continuous spraying technique has been successfully used to flameproof the surface of cotton pile rugs, napped blankets, and similar cotton products. This not only reduces significantly the chemical cost of flame retardants but also provides a more fluffy or bulky and resilient cotton product. (S2 1-190).

D. Cotton Textiles With Improved Luster

1. Processes for Imparting Durable Luster and Related Appearance Characteristics to Cotton Textiles. Cotton fabrics possessing the combined properties of permanent luster, excellent wash-wear characteristics, and extraordinarily high tearing strength have been developed in recent research. The process employed consists of weaving the fabrics from yarns mercerized under high tension and treating the fabrics with resins such as dimethylol ethyleneurea (DMEU). After as many as 35 launderings, the fabrics still retain considerable luster and extremely high wash-wear ratings. The luster obtained by weaving sateen from tension-mercerized yarn for the face of the fabric and unmercerized yarn for the back of the fabric was as great as the luster of sateen woven from tension-mercerized yarn for both warp and filling. This could reduce the cost of making a lustrous fabric.

The calendering without friction of DMEU-impregnated fabrics woven of pre-mercerized yarn, and the subsequent resin curing of these fabrics, produced wash-wear fabrics with high luster and sheen without the undesirable gloss obtained when friction-calendering was used. Luster of uncalendered fabric and also the effects of friction calendering remained durable after 20 or more standard launderings, although there was some decrease in shine.

Several textile finishing companies are evaluating lustrous fabrics produced in the research. Fabrics with durable luster and the other properties achieved are in great demand for many types of clothing and household items. (S2 1-194).

E. Stretch and Bulked Cotton Products

1. New and Improved Processes for Production of Stretchable Cotton Yarns and Fabrics Using Chemical and Mechanical Treatments. Stretchable cotton yarns and fabrics that retain their elastic properties after repeated use are being developed through the application of thermosetting resins and various mechanical treatments. Twist studies have established that for a 24/2 stretch-type cotton yarn of optimum properties produced by the back-twist method (DMEU resin) a single yarn twist multiplier of from 4.0 to 4.5 "Z" twist, a ply twist multiplier of 7.0 "Z" twist, and a 15.0 twist multiplier in the "S" twist direction for backtwisting should be employed. Stretch-type yarns were produced having over 200% elongation-at-break and were woven into fabrics having over 100% elongation-at-break in the filling direction. Filling-stretch fabrics from yarns produced by the back-twist method using either DMEU or a triazine-type resin have shown good durability to home and commercial-type laundering procedures. The fabrics showed no appreciable losses in resin content or strength and only slight decreases in stretch after 60 commercial launderings. Research to determine the effect of fabric structure on the stretch and other physical properties of filling-stretch fabrics has indicated that change in either ends or picks per inch is equally effective in controlling the fillingwise stretch of the fabrics. This work will be extended to two-way stretch fabrics. Optimum structures of stretch fabrics will be designed for specific end uses. (S2 1-193 (Rev.)).

Conventional twist and high twist yarns spun from highly acetylated cotton roving produced good stretch yarns by the falsetwisting and backtwisting processing techniques. It is indicated that significant stretch will be obtained in woven or knitted fabrics prepared from such stretch yarns. Also, basic information obtained on the relationship of polymer content to the amount of stretch that can be imparted to cotton yarn by cyanoethylation and graft polymerization of acrylonitrile, followed by backtwisting, has shown that this is a promising approach for making textured and stretch yarns. Other chemical and mechanical means for producing the crimped elastic cotton yarns will be investigated. (S2 1-213).

Initial experimentation by the contractor (Clemson Agricultural College) in research to develop cotton knit fabrics having increased bulk, warmth, and dimensional stability by application of finishing agents has shown that a vacuum impregnation-centrifugation method of application imparts bulk to the fabrics. The best methods for applying, drying, and curing several finishing agents, including DMEU, APO, and an epoxide, are being established. Durability, dimensional stability, and warmth measurements will be made and related to the bulk imparted to fabrics thus far prepared. (S2 1-205(C)).

Experimental work was initiated in contract research at North Carolina State College on evaluation of stretch-type cotton yarns in knit wear. The contractor has established satisfactory knitting conditions and techniques to produce knit fabrics from 24/2 and 60/2 stretch-type cotton yarns made by the back-twist method. The 24/2 yarns will be used to produce socks and the 60/2 to produce T-shirts. The garments will be evaluated in in-service tests. Knitting experiments are in progress on stretch yarns made by the false-twist method so that satisfactory knitting techniques and optimum fabric structures may be established for this type yarn. (S2 1-197(C) (Rev.)).

2. New and Improved Processes for Production of Stretchable Cotton Textiles Using Slack Mercerization and Other Type Swelling Treatments. An improved method for producing stretch cotton yarns by slack mercerization has been found. It consists of crosslinking the cellulose while swelled in a mercerizing caustic solution. This process yields yarns having more shrinkage than usual, thus imparting a higher degree of stretch. The wet crosslinking also improves the recovery properties of the yarn. Development of practical processes to crosslink the cotton cellulose in its most shrunken condition should permit use of normal washing and drying operations on the yarns without reduction of stretch properties as occurs when wet slack mercerized yarns are restretched during washing and drying. (S2 1-213).

Coordinated research between the Southern Division and knitters, weavers, and finishers has contributed significantly to the rapidly advancing commercialization of stretch cottons. At least twenty companies in the U. S. are in commercial production of all-cotton stretch fabrics by slack mercerization. Most of the production is going into apparel, but upholstery and slip cover materials are also produced. Several other domestic companies plan to market the all-cotton stretch fabrics, and these fabrics are also being commercially produced in Canada, Japan, and Europe. Slack-mercerized all-cotton stretch socks are in limited production by one company, and indications are that additional firms are planning commercial production. Industry is also interested in molding and coating stretch fabrics.

3. Resilient and Cohesive Cotton Batts from Low Cost Cotton. Continued good progress has been made in the research on cotton batting conducted in cooperation with the National Cotton Batting Institute, the Textile Waste Association, the National Cottonseed Products Association, and The Foundation for Cotton Research and Education. Batting products giving improved recovery from deformation loading at both 60% and 100% relative humidity have been produced using certain spray formulations containing more than one resin or latex. Studies of drying and curing of resin/latex treated batting indicated that the pressure drop through the spray damp batt is critical during the drying phase and must be maintained below 0.5" of water, whereas pressure drop during curing is not nearly so critical. Only very slight improvement in product performance has been achieved thus far through use of mechanical processing techniques for random orientation of fibers in the batting array. However, the fiber orientation studies will continue both on equipment fabricated at SU and on commercial type equipment for nonwovens.

Emphasis in recent work has been on obtaining practical data on formulations, drying and curing variables, and product performance improvement to speed commercialization of the new cotton batting. Early commercialization seems assured. Based on satisfactory pilot-plant evaluations which they conducted, two companies plan to purchase drying and curing ovens for full commercial production of the products. These companies have installed pilot lines for the production of the new batting, and eight additional companies are in some stage of installing pilot lines. Tests on auto seat cushions by two major automobile manufacturers has resulted in tentative approval of the batting products for use in the 1965 models. (S2 1-181 (Rev.)).

F. Effect of Yarn and Fabric Construction on the Physical Properties of Chemically Treated Cotton Fabrics

1. Effect of Fabric Structure on Properties of Chemically Treated Fabrics. Research has been initiated to investigate the effect of yarn and fabric structures on the properties of cotton stretch fabrics produced by slack mercerization with and without subsequent resin treatment. The work has already led to products with improved stretch and recovery properties. The amount of easy filling stretch in plain and twill weave fabrics (slack mercerized, resin treated) was increased by using filling yarns the same size or smaller than the warp yarns, and by employing filling yarns with higher twist. In the latter case the percent growth after stretching also increased. The research results should enable companies producing slack mercerized fabrics to improve the performance of their products. Research will continue on one-way stretch fabrics and be extended to two-way stretch fabrics. To adequately satisfy all the potential markets for stretchable cotton fabrics, it will be essential that fabrics with two-way stretch be developed. (S2 1-226).

The research to determine the feasibility and practicality of chemical tretament of cotton roving was terminated. A versatile unit was developed for batchwise chemical treatment of roving in package form; it operates under controlled conditions over a wide range of processing variables with minimum package deformation during processing. With suitable modifications the apparatus could be adapted to continuous operations. The operating techniques devised for the new apparatus are applicable to dyeing as well as to resin and other chemical treatments. (S2 1-184).

The experimental work on production of acceptable cotton crepe fabrics has been completed by the contractor (Philadelphia College of Textiles and Science). Based on experimental weavings of small yardages of fabrics from the crepe-type yarns developed, a selection of the most satisfactory cotton crepe was made, and fifty yards of this fabric were produced for evaluation. Although it is possible to produce satisfactory cotton crepes, the cost involved is so high it is unlikely they will be able to compete with rayon crepes of the same weight. (S2 1-157(C)).

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AREA NO. 6 - COTTONSEED PROCESSING AND PRODUCTS

Problem. Cottonseed products, currently approximately two billion pounds of oil and 2.5 million tons of meal derived from the annual domestic production of cottonseed, face increasing competition for markets. For its chief market, edible products, cottonseed oil must compete with other vegetable oils and animal fats. The nation's capacity for producing these oils and fats is so great that supplies can be expected to exceed both domestic and foreign demand for some time to come. Cottonseed meal, used chiefly as a protein supplement in feeding ruminant animals, faces serious competition from synthetic urea and other supplements. Improvements in the quality and utility of cottonseed oil are needed to retain present and open new markets for the currently large and possibly greater future production.

As an illustration, there is a discrimination in the markets against 25% to 50% of the current production of cottonseed oil due to the presence of reddish colors that are not removed by present commercial refining, bleaching and deodorizing methods. It is essential that information be developed on the chemistry of the pigments responsible for the off-colors, and that practical means be developed to eliminate them and thus upgrade the oils, particularly for use in margarine and shortening. Additional information is urgently needed on the chemical, physical, and biochemical properties of cyclopropene fatty acids in cottonseed and means of converting them, if found necessary, into physiologically inert forms. New types of modified fats, such as polyester and polymeric fats, need to be developed from cottonseed oil for applications in the fields of edible and inedible coatings, waxes, resins, plasticizers, and lubricants. Cocoa butter-like fats and other confectionery fats derived from cottonseed oil could also provide new markets for large quantities of oil. Fundamental information is needed on hydrogenation to permit production of improved plastic fats. Other areas in which markets for cottonseed oil need to be developed through research include fat emulsions for intravenous feeding, edible emulsifiers, and fatty acid amides and derivatives for use as plasticizers, plastic foams and other industrial products. Improvement in the quality and nutritive value of cottonseed meal is needed so that it can better compete with other protein feed supplements. Additional information is needed on the physiologically active constituents of the meal responsible for egg abnormalities, swine mortalities and growth abnormalities of young animals that limit cottonseed meal's usefulness in poultry and swine rations, and for the recently reported implication of cottonseed meal in the incidence of trout hepatoma which has resulted in its elimination from use in fish feeds in certain areas. Processing methods must be devised for the commercial production of meals that can be fed to broilers, laying hens and swine, safely and without restriction. In order to lay the necessary groundwork for advances in cottonseed research on food, feed and industrial products and processing technology, additional fundamental information is also needed

on the chemical composition and properties of cottonseed and of various cottonseed products.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving organic chemists, physical chemists, analytical chemists, biochemists, chemical engineers, physicists, and microbiologists engaged in both basic and applied studies on cottonseed and its products to develop new or extended uses for these materials.

Research to develop fundamental information on the chemical composition and properties of cottonseed products is conducted at New Orleans, Louisiana, as a basis for efficient applied research in the fields of food, feed, and industrial products from cottonseed. Some important phases of current work involve research on the chemical, physical and biochemical properties of cyclopropene fatty acids and other cottonseed constituents; and on fungi and toxic fungal metabolites which may develop in cottonseed and its processed products. The Foundation for Cotton Research and Education contributes towards research on the isolation and characterization of cyclopropene ring fatty acids of cottonseed. The National Cottonseed Products Association supports a Postdoctoral Research Associateship for conducting pioneering research on cottonseed and cottonseed constituents. Additional research on chemical composition and physical properties is carried out under contract at the University of Tennessee, Knoxville, Tennessee, on investigations of gossypol esters and mild oxidation products of gossypol and gossypol derivatives; at the University of Illinois, Urbana, Illinois, on investigation of the chemical and physical properties of cyclopropene fatty acids in cottonseed; and at Purdue Research Foundation, Lafayette, Indiana, on fundamental investigations of chemical transformations of olefinic compounds of fats and other agricultural materials by hydroboration and subsequent reactions to develop basic information for the production of useful products.

New and improved food products and processing technology are developed in research conducted at New Orleans, Louisiana. Methods are sought to produce improved cottonseed oils, and confectionery fats, polyester products, and fat emulsions for intravenous nutrition from cottonseed oil. The research on confectionery fats is cooperative with the National Confectioners' Association who maintain a Fellowship at the Southern Regional Research Laboratory, New Orleans, Louisiana, in partial support of the work, and evaluate promising research products. The Office of the Surgeon General supports the research on fat emulsions. This research is conducted cooperatively with the U. S. Army Medical Research and Nutrition Laboratory and several medical school research groups. Informal cooperation is maintained with industry in connection with the research on new and improved food products and processing technology. Additional research on new and improved food products and processing technology is conducted under contract at the University of Illinois, Urbana, Illinois, on chemical investigations of

cyclopropenoids to develop practical means of eliminating or physiologically inactivating the cyclopropenoid constituents of cottonseed oil.

Research is carried out at New Orleans, Louisiana, to develop new and improved feed products and processing technology for cottonseed. Investigations are in progress to isolate and identify the physiologically active constituents in cottonseed meals that adversely affect the utilization of the meal as a protein supplement in nonruminant feeding. Animal tests in connection with the overall research program are conducted through the cooperation of nutritionists in State Agricultural Experiment Stations and the Animal Husbandry Research Division. The Pharmacology Laboratory at the Western Regional Research Laboratory, Albany, California, cooperates by conducting small-animal studies to determine the physiological and pharmacological effects of cyclopropene acids and toxic fungal metabolites. In research directed toward providing a basis for the ultimate commercial production of cottonseed meals that can be fed to swine and poultry without restriction, as well as to ruminant animals, cooperation is maintained with the National Cottonseed Products Association, members of the cottonseed industry, and nutritionists in public and commercial agencies. Additional research in the field of new and improved feed products and processing technology is in progress under contract at IIT Research Institute, Chicago, Illinois, on development of practical processing methods for inactivation of cyclopropene groups in cottonseed meal that decrease its value as a feed for laying hens.

Research to develop new and improved industrial products and processing technology is conducted at New Orleans, Louisiana. Present emphasis is on amide derivatives of long-chain fatty acids. Informal cooperation is maintained with industrial firms for the evaluation of promising research products for specific end uses. Additional research on new and improved industrial products is being carried out under contract at the University of Arizona, Tucson, Arizona, on the polymerization of reactive chemical intermediates derived from cottonseed oil and other agricultural materials to produce polymers having potential industrial utility; and at U. S. Industrial Chemicals Co., New York, N. Y., on copolymerization of ethylene with unsaturated fatty acids and other selected derivatives of agricultural materials to extend their utilization in commercial plastics.

Other research on chemical composition and physical properties is in progress under grants of P.L. 480 funds to the following foreign institutions: British Food Manufacturing Industries Research Association, Leatherhead, Surrey, England, for fundamental studies of the fatty acid and glyceride composition of cottonseed oil and the crystallizing behavior of some of the major components (project duration - 4 yrs.); University of Bombay, Bombay, India, for a study of the relationship of substituent fatty acid groups on the physical properties of diacid triglycerides of palmitic and stearic acids as a means of increasing the utilization of cottonseed oil for food and industrial purposes (project duration - 5 yrs.); Israel Institute of Technology, Haifa, Israel, for investigation of π-complexed organometallic

compounds derived from polyunsaturated fatty acids to obtain fundamental information needed in expanding the utilization of cottonseed oil (project duration - 5 yrs.); University of Rome, Rome, Italy, for basic investigations on the physical and physicochemical properties of cottonseed proteins (project duration - 5 yrs.); and Commonwealth Scientific and Industrial Research Organization, Ryde, Australia, for an investigation of the chemistry and biological effects of cyclopropenoid compounds that occur in cottonseed and its products (project duration - 5 yrs.).

Additional research in the field of new and improved feed products and processing technology is in progress under a grant of P.L. 480 funds to Instituto Farmacologico "Mario Negri", Milan, Italy, for a study of the mechanism of gossypol toxicity counteraction by L-lysine (project duration - 5 yrs.).

Additional research to develop new and improved industrial products and processing technology is in progress under grants of P.L. 480 funds to the following foreign institutions: University of Montevideo, Montevideo, Uruguay, for research on the preparation, characterization, and evaluation of derivatives of gossypol for use as biologically active materials, ultraviolet absorbers, and other products (project duration - 5 yrs.); Indian Institute of Science, Bangalore, India, for studies of the addition of carbenes to unsaturated fatty materials derived from cottonseed oil to provide possible new outlets for utilization of the oil (project duration - 5 yrs.); and National Chemical Laboratory, Poona, India, for investigation of the synthesis and properties of new-type glycol mono alkyl ethers for control of water evaporation to extend the industrial utilization of cottonseed oil (project duration - 5 yrs.).

The Federal in-house scientific effort devoted to research in this area totals 50.9 professional man-years. Of this number 20.4 is devoted to chemical composition and physical properties, 17.2 to new and improved food products and processing technology, 9.6 to new and improved feed products and processing technology, and 3.7 to new and improved industrial products and processing technology. The contract research involves an additional 6.9 man-years, 2.4 being on chemical composition and physical properties, 0.9 on new and improved food products and processing technology, 1.9 on new and improved feed products and processing technology, and 1.7 on new and improved industrial products and processing technology. P.L. 480 research involves 9 grants, of which 5 are on chemical composition and physical properties, 1 on new and improved feed products and processing technology, and 3 on new and improved industrial products and processing technology.

The following lines of work were terminated during the year: (1) Engineering studies to develop a commercial process for preparing cocoa butter-like fat from cottonseed oils (under new and improved food products and processing technology); and (2) Pilot-plant development of a cottonseed extraction process using hexane-acetone-water solvent mixtures to a stage suitable for commercial evaluation (under new and improved feed products and processing

technology).

PROGRAM OF STATE EXPERIMENT STATIONS

Station research on cottonseed utilization is directed toward increased feed use. When protein quality is poor, usually the lysine in the protein has combined chemically with sugars, with fatty materials or, in the case of cottonseed, with gossypol. Studies designed to determine the extent to which lysine has reacted and to better understand the reactions which interfere with proper protein utilization are in progress. High quality proteins in the rations of swine and poultry decrease costs. As a result of this emphasis, the chemical properties and biological significance of gossypol-protein complexes are of major concern. Experiments designed to elucidate the effects of proteolytic enzymes on gossypol-protein complexes have revealed that trypsin treatment results in liberation of more than half of the gossypol as gossypol-peptide compounds. To date no enzyme has been found that will split the Schiff base type of linkage between gossypol and the epsilon amino group of lysine in the protein. This study is continuing and attempts are being made to determine the sequence of the amino acids in the peptide-gossypol compounds.

Other studies include work on developing suitable methods and techniques for determining harvest-aid chemicals or metabolites that may remain as residues in cottonseed. The action of gossypol at various concentrations on the in vitro activity of selected enzymes and enzyme systems is being investigated. Investigations on the influence of gossypol in swine rations are continuing and have revealed that gossypol is eliminated at a rather slow rate from the liver and spleen of the pig. Other related research centers on determining the nutritional value of cottonseed meal in animal rations.

The total research on the utilization of cottonseed amounts to approximately 1.7 professional man-years.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Chemical, Physical, and Biological Properties and Structural Factors of the Proteins. The composition, properties, structural factors and reactions of oilseed proteins and associated materials are being investigated in a program of pioneering research conducted by the Seed Protein Pioneering Research Laboratory. The fundamental information developed should lead to new concepts and possibly new applications for oilseed proteins, including cottonseed protein. Since peanuts were found to be an especially suitable experimental material and employed for much of the early pioneering research on seed proteins, the report of progress in the research is given in Area No. 7, "Peanuts Processing and Products," as in the previous report.

Substantial progress has been made in a basic investigation of the physical and physicochemical properties of pure isolated cottonseed proteins under a P.L. 480 grant at the University of Rome. A monodisperse major protein component has been isolated from the protein extracted from a glandless variety of cottonseed. Studies are underway on the chemical and physicochemical characterization of this protein fraction. Work under this project is expected to yield new and fundamental information concerning the nature of cottonseed proteins and the enzymes of the resting cottonseed in relation to amino acid metabolism. Such information is needed in the application of cottonseed proteins to human food needs. (UR-E15-(40)-33).

2. Chemical and Physical Properties of Pigments and Minor Constituents Including Cyclopropene Fatty Acids. In the contract research at the University of Tennessee on gossypol esters and mild oxidation products of gossypol and its derivatives, two crystalline products have been isolated from the early stages of alkaline hydrogen peroxide oxidation of gossypol. One of these contained ethyl alcohol of crystallization. Tentative structures for these products have been proposed. Other work has resulted in the discovery and identification of a highly colored, purple-brown hydroxyquinone pigment formed by oxidation of gossypol under very mild conditions (by the action of limited amounts of oxygen on aqueous alkaline solutions of gossypol). The quinone is purple in alkaline solutions and red in acidic solutions. It is not alkali-fast, and the red color it imparts to cottonseed oil fades on exposure of the oil solution to light. The chemical properties of the quinone will be investigated. The information being developed will be of value in research on the problem pigments of off-colored cottonseed oils and on the problem of the metabolic fate and physiological properties of gossypol in animals ingesting cottonseed products. (S4 1-103(C)).

Extensive fundamental investigations of the cyclopropene fatty acid constituents of cottonseed oil have continued along several lines in in-house research. Several new procedures for removing or inactivating these constituents have been developed. Heat treatment of the oil with certain acids, including cottonseed acids, capric acid, citric acid, oxalic acid and formic acid, was employed in processes for production of Halphen-negative oils. Halphen-negative oils prepared by these chemical procedures will be fed to laying hens to determine physiological effects on egg quality.

Only limited success has been achieved in further purification of methyl sterculate by crystallization procedures. Maximum purity of product was about 83 percent. However, N-benzylsterculamide -- a crystalline cyclopropenoid which is solid at room temperature -- was prepared in 87% purity. Repeated solvent crystallization of methyl esters of cottonseed oil and Hibiscus syriacus has yielded products containing 15% and 25% methyl malvalate, respectively. Low-temperature crystallization techniques were scaled up in pilot plant experiments to produce sizeable quantities of cottonseed methyl esters rich in malvalate (about 10% concentration) for possible use in animal feeding tests and other phases of the cyclopropene research program. Procedures for obtaining still higher concentrations of

the cyclopropenoids will be sought.

In further work on analytical procedures for cyclopropenoid fatty acids, a method applicable to the determination of these acids in rancid cottonseed oils was developed. The stepwise hydrogen bromide titration procedure also can be used as a means of following the autoxidation of methyl oleate or glycerides, and will afford a useful new tool for investigation of the mechanism of autoxidation. By applying previously developed analytical methods to the analysis of crude oils from 25 species and varieties of cottonseed, it was found that these oils ranged from 0.56 to 1.17% in cyclopropene fatty acid content (calculated as malvalic acid). The development of analytical techniques for cyclopropene fatty acids, particularly micro or semimicro methods and a method applicable to meals, will be continued. (S4 1-105).

3. Chemical, Physical, and Physiological Properties of the Oil, Fatty Acids, and Derivatives. Feeding tests have been continued in an effort to ascertain whether the presence of physiologically active cyclopropene fatty acid constituents in cottonseed oil constitutes a real problem from the standpoint of utilization of the oil for food uses. Considerable data have been accumulated concerning the effects of ingestion of cottonseed oils containing various amounts of cyclopropenoids on the fatty acid composition of various tissues and organs of experimental animals but, generally, firm conclusions cannot yet be drawn. The tissues from rats fed different fractions of cottonseed oil at the Pharmacology Laboratory, WU, are currently being analyzed for fatty acid distribution. In addition, other tests have been designed to determine whether Halphen-negative oils incorporated into the diets of laying hens have physiological effects on egg quality. For one test, now completed, commercially refined oils were bleached with alumina treated with sulfurous acid to remove their response to the Halphen test; the eggs produced were normal with respect to distribution of fatty acids in yolk fats and the color and pH of yolks and whites. Other experiments are in progress to confirm these findings. Halphen-negative oils prepared by other chemical procedures are also being used in similar feeding tests on hens. (S4 1-105).

Further research has been conducted under contract at the University of Illinois on chemical and physical properties of cyclopropene fatty acids in cottonseed. Investigations of the Halphen reaction of methyl sterculate indicated that a red compound, $C_{20}H_{36}O_2S_2$, is one of the substances formed. One sulfur atom is eliminated upon reduction of this compound with zinc. Reduction of the mixed methyl esters from the seed oil of Sterculia foetida with lithium aluminum hydride yielded a mixture of hydrocarbons that was not resolved into its component parts. A 50% yield of sterculene was obtained when the reduction was carried out with pure methyl sterculate. Various chemical intermediates to model compounds for use in studies of the cyclopropene ring have been synthesized. Trans-2-phenylcyclopropyltrimethylammonium iodide was prepared for making the corresponding phenylcyclopropene compound. The expected cyclopropane was not obtained by reaction of

benzylethylene oxide and triphenylcarbethoxymethylene phosphorane. Effort is also being made to develop better methods for isolation of cyclopropene fatty acids. Countercurrent distribution of methyl esters from an oil sample low in methyl sterculate gave some enrichment in methyl sterculate, but separation was incomplete. Urea clathration remains the superior method for isolation of sterculic acid and its methyl ester. The fundamental information being developed should provide a basis for improving analytical methods for cyclopropene acids and facilitate development of improved procedures for removing or inactivating the cyclopropenoids of cottonseed oil.(S4 1-104(C)).

In resumed work on investigations of solubilities of long-chain fatty acids and their derivatives, additional solubility data for the pure cyclohexylamine salts of capric, heptadecanoic, and elaidic acids in methanol were obtained to complete these solubility curves. A number of additional methods of correlation and prediction of solubilities of fatty acids and derivatives (not necessarily homologs) have been worked out theoretically from thermodynamic principles and tested sufficiently to show their feasibility. It is proposed to complete the development of and make use of these and additional methods under a new research project. (S4 1-88).

Fundamental information on the chemical transformation of olefinic compounds of fats by hydroboration and subsequent reactions has been developed in contract research initiated at Purdue Research Foundation. Organoboranes were synthesized from olefinic ethers, and the resulting alkoxyorganoboranes were coupled to produce the respective dimers. 1,8-Dimethoxyoctane, 1,22-dimethoxydocosane, 1,8-diphenoxyoctane and 1,22-diphenoxydocosane were prepared from 4-methoxybutene, 11-methoxyundecene, 4-phenoxybutene and 11-phenoxyundecene, respectively. Preliminary experiments were conducted with olefinic esters, and 1,22-docosanedioic acid was prepared from methyl 10-undecenoate. The systematic study of hydroboration of olefins containing various functional groups will continue. The research results should provide a basis for preparation of potentially useful products from fatty acid olefinic compounds. (S4 1-112(C)).

In P.L. 480 research at the British Food Manufacturing Industries Research Association, studies are in progress on the fatty acid and glyceride composition of cottonseed oil and the crystallizing behavior of some of the major components. The fatty acid composition of a number of oils from cottonseed of various origins and processing histories have been examined by several different methods. Gas-liquid chromatography yielded results nearest to the accepted true values. Fractionation by low temperature crystallization has indicated that, although cottonseed oil contains 3 major component fatty acids, only 4 out of 26 probable triglycerides occur to the extent of over 8%, and the minor component acids are very uniformly distributed throughout the glyceride components of the oil. Additional information is being sought through the application of techniques such as countercurrent distribution, thin layer chromatography and lypolysis employing lipases. Further progress in the research is expected to provide data useful in the selection and processing of cottonseed oils for the commercial production of improved salad oils in optimum yields. (UR-E29-(40)-26).

In P.L. 480 research at the University of Bombay, studies are being made of the relationship of the substituent fatty acid groups to the physical properties of diacid triglycerides of certain saturated fatty acids, including those that occur normally in cottonseed oil. The diacid triglycerides that are of interest in this work are those containing one or two molecules of palmitic or stearic acid, and two or one of even-carbon saturated fatty acids of the series from acetic to stearic acid. Pure 1,3-diglycerides have been prepared and work is underway to prepare pure 1,2-diglycerides, from which the corresponding diacid triglycerides are synthesized. Progress is being made in determining the properties of the synthesized diacid triglycerides and their binary mixtures. The information obtained is expected to provide the basis for the further development of fats and oils specifically tailored for special food and industrial end uses. (UR-A7-(40)-3).

Research is just getting underway under a P.L. 480 grant at the Israel Institute of Technology (Technion) in which a basic investigation is being made of π complexed organometallic compounds derived from polyunsaturated fatty acids that occur in cottonseed and other fats and oils. Progress in the initial stages of the work has been made in applying the reactions to certain model compounds to develop techniques for extending the reactions to the polyunsaturated fatty acids. It is expected that the information obtained in later stages of the research will indicate opportunities for increasing the use of fatty acids from cottonseed and other oils through the development of new or expanded industrial applications. (UR-A10-(40)-34).

4. Investigation of Occurrence,Determination, and Properties of Fungi and Toxic Fungal Metabolites Which May Develop in Cottonseed and Its Processed Products. Research was recently initiated to isolate, identify, evaluate, and control the fungi and toxic fungal metabolites which may develop in cottonseed and its processed products. The importance of the potential problem of various agricultural commodities becoming contaminated with toxic microbial products is receiving increasing recognition.

Major emphasis in initial work has been on development of basic analytical methodology. The unique pigmentation problems in cottonseed meals and meats necessitated the development of a new analytical method for determination of aflatoxin in these products. Extraction solvents and procedures for removing interfering gossypol pigments from the extracts were studied to devise suitable procedures. The lower limits of detection of aflatoxin are presently being explored. In other experiments, a sample of deteriorated cottonseed (acid delintered) was found to be contaminated internally with Penicillium notatum, a mold in the P. oxalicum series, and a Cladosporium sp. Organisms found in the whole, crushed seed were A. elegans, 2 species of Penicillium, A. niger, A. awamori, A. niger gr., Rhizopus nigricans, and A. amstellodami.

Several selected commercial cottonseed meals were exhaustively extracted successively with ethyl ether-ethanol and ethyl ether-ethyl acetate to obtain extracts for use in cooperative studies on trout hepatoma with the

Western Fish Nutrition Laboratory, U. S. Department of Interior, and others. Attempts are being made to determine if the extracted components -- whether inherent in or adventitious to the meals -- contribute to the incidence of liver cancer in hatchery fish. The possibility exists that mold contamination of trout diets could be responsible for the hepatomas. (S4 1-116).

B. New and Improved Food Products and Processing Technology

1. New Edible Oil Products Including Confectionery Fats, Food Coatings and Other Specialty Products. A process has been developed for preparation of an improved cocoa butter-like fat by the direct esterification of the diglycerides of palmitic and stearic acids with oleic acid. It was found that most of the approximately 20% of unwanted, high-melting fats formed during the esterification can be removed by a relatively simple fractionation (crystallization) process. The cocoa butter-like fat product has the properties desired in a confectionery fat, including a short melting range, brittleness at room temperature, and good compatibility with cocoa butter. Larger quantities of the product will be prepared for evaluation. A commercial propylene glycol monostearate was also successfully esterified with oleic acid. Although the esterified product has too low a melting range (5°-15° C.) to be used as a cocoa butter-like fat, it may have other confectionery fat uses. Further work will be conducted on the direct esterification process.

Additional research on the previously developed method for rapid tempering of fats showed that increasing the orifice diameter of the homogenizer from 1/16 inch to 1/8 inch did not change the amount of mechanical working necessary to temper super-cooled, liquid cocoa butter. Both milk chocolate and chocolate liquor were successfully tempered by mechanical working, temperatures between 25° and 30° C. proving best. Satisfactory results were obtained by a commercial concern in test pilot-plant runs to evaluate the rapid tempering process for confectionery coatings. The research on confectionery fats is supported in part through a Fellowship sponsored by the National Confectioners' Association. (S4 1-125, Pending).

In recent research to develop new polyester products from cottonseed oil, emphasis has been placed on processes involving direct esterification of amylose with fatty acids. Further examination of products prepared by simple catalytic esterification of amylose with palmitic acid using dimethylsulfoxide as solvent revealed that the solvent entered the reaction and sulfur-containing esters were present in the products. This would make these particular products unsuitable for edible uses. A partially acylated amylose was prepared by dissolving the amylose in dichloroacetic acid and esterifying with fatty acid in hexane solution under conditions which remove water of esterification as it is formed. Research will be continued to establish optimum conditions for esterifying amylose in dichloroacetic acid solution. Amylose esters of long-chain fatty acids have attracted some industrial interest; and development of suitable methods for their preparation should facilitate their introduction and utilization, especially in

food coatings. A commercial firm is currently evaluating samples of the acetate, palmitate, and stearate esters of amylose as edible coating systems for freeze dried foods.

In further investigations of fungistatic activity of fatty acids and fats, experiments using the paper disc test method showed that growth of all types of molds studied was inhibited by caproic acid and tripropionin; growth of four Phycomycetes was retarded by trinonanoin, tridecanoin, triundecanoin, and triundecenoin; and growth of Rhizopus nigricans was temporarily prevented by triundecanoin and triundecenoin. (S4 1-90).

Improved fat emulsions for intravenous nutrition have been developed in research supported by the Office of the Surgeon General and conducted cooperatively with the U. S. Army Medical Research and Nutrition Laboratory and several medical school research groups. Animal tests of the individual emulsifiers of the previously developed SR emulsion 695 indicated all to be physiologically nonreactive if purified correctly. Modified emulsion 695 (reduction in emulsifier concentration) also was physiologically satisfactory. SR 695 or its modified form is ready for commercial preparation, and further development work on these particular emulsions is not contemplated at this time. New type, physically stable emulsions of cottonseed oil and of soybean oil have been formulated employing pure egg lecithin as the sole emulsifier. This was made possible by the successful development of a more efficient and higher yielding chromatographic method for isolation of the lecithin. The physical stability of the new emulsions is satisfactory at a 1 wt. % concentration; a pH of 6.6-6.8 is optimum. An isotonic solution of glycerol is employed as the aqueous phase in these emulsions. The lecithin-stabilized emulsions had no adverse effect on blood pressure of test animals. Extensive physiological evaluation of a soybean oil emulsion (SR 151) in animals is now in progress at three cooperating institutions; a cottonseed oil emulsion (SR 152) will be evaluated in the near future. (SU-0-0-2(SG)).

2. Processing Technology Related to Improved Oil Products, Including Modifying or Eliminating Cyclopropene Acids in Cottonseed Oil. Completed engineering studies have laid the basis for and indicated the feasibility of developing a laboratory interesterification process for preparing cocoa butter-like fats into a commercial scale process. The effect of additional modifiers on shortening crystallization times in the various processing steps of the conventional two-solvent (acetone-hexane) process were investigated in recent bench-scale experiments. Palmitic acid gave the best yields for separating crude cocoa butter-like fats from fat-acetone miscella, but the yields were not as high as those previously obtained when the saturated fats separated from the first crystallization were used as a modifier. None of the modifiers investigated for accelerating the separation of the crude cocoa butter-like fats gave yields comparable to those obtained using the conventional long holding times. In tests of the second crystallization phase of the process employing a scraped-surface crystallizer, it was established that filtration rates of the acetone-fat slurries quick-cooled in the

crystallizer can be increased by increasing retention time after cooling beyond four minutes. In the purification step, employing hexane, a minimum cooling time of 90 seconds was found necessary to obtain a slurry whose filtration rate does not adversely change with increased holding time. A one-solvent (hexane) process for producing cocoa butter-like fats was also investigated but proved unsatisfactory. (S4 1-101).

A practical batch process for bleaching off-colored cottonseed oils with activated alumina has been developed through the pilot-plant stage. Test runs in the 200-pound batch pilot plant gave results closely comparable to those obtained in the smaller bench-scale tests. A commercially available alumina, quoted at 10.5 cents per pound, was employed at a dosage of 4% in the pilot plant experiments. A major producer and processor of cottonseed oil has conducted engineering cost analysis of the process and has scheduled plant-scale tests for the near future. Recent research demonstrated the regenerability of the commercially available alumina proposed for the "in plant" testing, thereby removing the last technical obstacle to commercial employment of the bleaching process.

Work is in progress to develop methods for upgrading the quality of cottonseed oils by improving the color and eliminating undesirable components such as cyclopropene acids. Alumina bleaching followed by steam deodorization reduced the malvalic acid content of a color-reverted oil from 0.59% to 0.08%, and reduced oil color from 8.5 red to 2.9 red. Treatment of the same oil with alumina gel, precipitated on bleaching earth with sulfurous acid, gave a Halphen-negative oil but had no significant bleaching effect. Use of activated alumina and alumina gels in combination with other chemicals will be investigated. (S4 1-92, S4 1-114).

The contractor (University of Illinois) has made good progress on chemical investigations of cyclopropenoids as a basis for developing practical means of eliminating or inactivating these type constituents present in cottonseed oil. The cyclopropenoid -- 1,2-diethylcyclopropene -- was synthesized by the photolysis of diazomethane in the presence of 3-hexyne. It has been isolated in pure form and characterized. Three of the major pigmented components of the reaction of this compound with a solution of sulfur in carbon disulfide (Halphen-test reaction) have been isolated in chromatographically pure form; one of the components has been identified as cis-1,5-diethyl-2,4-dithiabicyclo-(3.1.0)-hexane-3-thione. This is the first Halphen-test reaction product that has been isolated and identified. It lends support to the view that the mode of action of cyclopropenes in living organisms may be by way of reaction of cyclopropenes with SH sites of enzyme systems. This could afford a satisfactory explanation of the observed unusual physiological activity of cyclopropenoids. Partly as a result of this, research on the effect of cyclopropenes on specific purified SH-containing enzymes has already been initiated in the Seed Protein Pioneering Research Laboratory. (S4 1-107(C)).

C. New and Improved Feed Products and Processing Technology

1. Basic Research to Improve Nutritive Value of Cottonseed Meal for Poultry and Swine, Including Investigations of Physiologically Active Constituents. As a basis for further improvement of the protein quality of cottonseed meals, research investigations are in progress to obtain a thorough understanding of the composition of cottonseed proteins and their location anatomically. The hypocotyl and cotyledon of the cottonseed have been found to differ in their amino acid patterns; the hypocotyl is higher in lysine, threonine, isoleucine and leucine, and it is lower in arginine, aspartic and glutamic acid. The evidence that amino acids important to nutrition of animals are more concentrated in the hypocotyl may open the way for further improving protein quality of cottonseed products. Aqueous extracts (deionized water) of the hypocotyl and cotyledon show similar electrophoretic patterns, but only one band in these patterns shows characteristics similar to the five major bands noted on electrophoresis of the protein bodies of the cottonseed. The proteins of the protein bodies are generally characterized by low lysine and high arginine and glutamic acid contents. Analyses to determine the amino and fatty acid patterns of members of the genus Gossypium were carried out and much of the work has been completed. Preliminary data indicate that the amino acid pattern noted for seed from upland cotton may be characteristic of the genus.

Investigations to identify the factors in cottonseed meal that cause mortalities among swine have continued. Feeding tests with swine to locate lethal cottonseed meals have been completed. With three meals, mortalities varied from 87% to 100%. Commercial hexane-extracted meals (containing about 1% residual oil) caused 100% mortalities. No toxicities were noted in the case of the mixed solvent meals (meals extracted with acetone-hexane-water mixture) and, moreover, shoats fed these meals outgained those fed a soybean meal selected for comparison by the National Soybean Council of America. Comparison of a meal causing 100% mortalities in swine with a mixed solvent meal has been made in recently completed rat feeding tests at the Pharmacology Laboratory, WU. Tissues from the animals are being analyzed at the Southern Division. Feeding tests with laying hens seem to indicate that abnormalities in the fatty acid distribution in egg fat are associated with constituents in cottonseed oil. Various cottonseed pigment gland fractions, separated on the basis of solubility, were fed to laying hens in cooperative experiments with Ralston Purina Co. to determine physiological effects of different forms of bound gossypol. These fractions, as well as gossypol, caused decreases in egg production, egg size, feed intake and weight of hens, as well as egg normalities in yolk pH, pink whites and yolk discoloration. Examination of meals and of tissues from animals fed cottonseed products will be continued. (S4 1-110).

2. Processing Technology Directed Toward Improving Meals. The pilot-plant development of the mixed solvent (acetone-hexane-water) extraction process for cottonseed is essentially complete and the process is now considered ready for evaluation in commercial plants. The process can be easily adapted to existing basket extraction plants without additional extraction facilities if 1% residual lipids in the meal is satisfactory. Auxiliary

immersion extraction equipment would be needed to reduce residual lipids to about 0.5%. A horizontal immersion extractor with an inclined draining section, to replace the previously used inclined extractor, has been designed, installed and test operated satisfactorily. In pilot-plant tests, commercial-type equipment and processing conditions for desolventizing oil and meal gave satisfactory results in the mixed solvent process. Preliminary evaluation of capacitance instruments indicated that they can be successfully used in controlling the mixed solvent composition to the values selected, and in automatically controlling the solvent recovery and remake operations. The crude miscella from the mixed solvent extraction process has been miscella refined in a batch process to produce a prime bleachable oil, with nearly complete recovery of neutral oil. A typical plant layout and flow sheet for the new extraction process has been prepared, and cost estimates have been developed for new plants and for converting existing plants to mixed solvent extraction for capacities of 200 and 400 tons of cottonseed per day.

Two 1-ton lots of cottonseed meal having residual lipids contents of 0.7% and 1.0%, low free and total gossypol contents, and high available lysine contents were produced in the pilot plant by the mixed solvent process for use in feeding tests with swine, broilers, and laying hens. In swine feeding tests, these meals have equaled or exceeded soybean meal and greatly exceeded commercial cottonseed meals in performance. Excellent results were also obtained in feeding broilers, and additional tests are underway on broilers and laying hens.

New research will be initiated to investigate the processing characteristics of glandless cottonseed, and the refining and bleaching of cottonseed miscellas and oils of high gossypol content. (S4 1-111).

A study of rates of extraction of the oil of cottonseed with acetone-hexane-water solvent mixtures, and the properties of the marcs and miscellas, has been initiated to provide information basic to the production of processed cottonseed products of the highest quality. It was demonstrated that a very rapid and substantially complete extraction of oil from raw, moist cottonseed flakes can be achieved with acetone-hexane-water azeotrope in a four-step countercurrent extraction if light pressing (ca 100 psi for about 40 seconds) is employed between each pass in the extraction. Extraction time was about 3 minutes, and residual oil in the air dried marc was approximately 0.1%. The solvent-damp marc tends to form a plastic mass when it is kneaded in desolventization operations. The compositions of miscellas and properties of the marcs obtained by the azeotropic extraction will be investigated. (S4 1-123).

D. New and Improved Industrial Products and Processing Technology

1. Basic Research to Develop New Reactions and Products Suitable for Industrial Use. Additional N-disubstituted fatty amides have been prepared, characterized, and evaluated as polyvinyl chloride plasticizers. They will

be submitted for screening evaluations for antimycotic activity. The N-dialkylamides, a new class investigated, proved particularly interesting since preliminary results indicated their thermal stability was much better than that of most of the amides so far investigated, and they gave exceptionally low brittle points. Recent work on this class of amides -- specifically N,N-dialkyloleamides -- has indicated that as the size of the alkyl group increases, the volatility loss decreases. Brittle points were lowest for the n-butyl derivative (-63° C.) and the n-amyl derivative (-61° C.); beyond the hexyl derivative, the dialkyloleamides are incompatible. The n-butyl derivative has a markedly lower volatility loss, and gives a lower brittle point than the isobutyl derivative. It is comparable to dioctyl sebacate, a commercial plasticizer, which gives a brittle point of -65° C. The effect of acid moiety on properties of the N-dialkylamides will be investigated.

Plasticizer performance of the N,N-bis(2-methoxyethyl)- and/or N,N-bis(2-ethoxyethyl)amides of a number of fatty acids (including palmitic, stearic, oleic, linoleic, erucic, epoxystearic, dimer acids, parsley seed acids, rapeseed acids, L. douglasii acids, and selectively hydrogenated cottonseed acids) was also determined. All except the linoleic acid derivative exhibited good compatibility in vinyl chloride plastics. The N,N-bis(2-ethoxyethyl)stearamide is apparently the first compatible plasticizer containing the stearic acid moiety that has been found. In general, these amides are more efficient than dioctyl phthalate (DOP) and most of them, including the amides of the seed oil acids, give lower brittle points (-45° to -57° C.) than DOP (-33° C.). Many are thus comparable to dioctyl adipate (-55° C.) without the objectionably high volatility loss of the latter. The N,N-bis(2-ethoxyethyl)amides of epoxystearic and dimer acids gave lower brittle points than other amide derivatives of these acids so far studied, and the epoxy derivative exhibits a thermal stability equal to that of DOP and can be used as a compatible stabilizer. The N-dialkoxyalkylamides, as a class, appear to have good potential as specialty low-temperature plasticizers for polyvinyl chloride. Selected ones will be evaluated as softeners for nitrile rubber.

Based on earlier Southern Division research on piperidides of long-chain fatty acids, a chemical company is planning pilot-plant scale preparation of one of these plasticizers for test marketing. (S4 1-99, S4 1-124).

Further progress has been made in P.L. 480 research at the University of Montevideo, Uruguay, in the preparation, characterization and screening of a number of derivatives of gossypol having potential industrial utility. Several new imino derivatives (anils) have been prepared. Reduction of these to substituted amines by catalytic hydrogenation at moderate pressures has not been successful. However, use of higher pressures, or reduction by means of agents such as lithium aluminum hydride or sodium borohydride, is expected to yield derivatives having ultraviolet screening or antioxidant properties. Derivatives that have been prepared by reaction with hydantoin would be expected to exhibit physiological activity or catalytic activity

in certain organic reactions. Two classes of gossypol derivatives with carboxyl groups in the molecule have also been prepared. These compounds may have fungicidal or germicidal properties. Screening for such potential uses is being considered. (UR-S9-(40)-2).

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AREA NO. 7 - PEANUTS PROCESSING AND PRODUCTS

Problem. Peanuts constitute a major cash crop in the Southern States and are in surplus. Because of the high price of peanuts in the United States, peanuts are used almost exclusively (approximately 73 percent of the crop) in foods such as peanut butter, confections, and roasted and salted nuts. New type food products and improvement in the quality and uniformity of existing products are needed to increase consumer acceptance and extend markets; the average per capita consumption has been rather stable since World War II. The increased trend toward mechanical harvesting has necessitated the use of artificial means for curing and drying peanuts, with the result that processed peanuts and peanut products do not always possess the same desirable flavor and physical properties as peanuts which have been cured slowly in the field. Information is needed as to the physical and chemical characteristics of those chemical constituents in peanuts which affect the properties of processed products as a basis for developing new or improved products and processing procedures. Fundamental studies of peanut protein and associated materials could similarly lead to the development of new concepts and new uses. Additional information is urgently needed on the isolation, identification, evaluation, and control of mold toxins such as aflatoxin in peanuts and processed peanut products.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving organic chemists and biochemists, engaged in basic studies on peanuts and peanut products to increase consumer acceptance and extend markets for peanuts.

Research to develop basic information on the chemical composition and properties of peanuts, its constituents, and processed peanut products is carried out at New Orleans, Louisiana. As a part of the Seed Protein Pioneering Research Laboratory's research on various seed proteins, fundamental investigations of peanut proteins and associated materials are conducted to form the basis for developing new concepts and perhaps new uses for peanuts and peanut proteins. In other in-house research, peanut constituents and their modification by processing that influence nutritive properties and consumer acceptance of processed peanut products are studied. Current phases of this research include investigations of the proteins and nonglyceride lipid-soluble constituents of peanuts and processed peanut products; and isolation, identification, evaluation and control of fungi and toxic fungal metabolites which may develop in peanuts and its processed products. The Crops Research Division of ARS, the Agricultural Marketing Service, and several State Experiment Stations cooperate in the research by providing samples of peanuts of known variety and history. The Pharmacology Laboratory at the Western Regional Research Laboratory, Albany, California, and the Food and Drug Administration cooperate in certain biochemical aspects of the research.

Additional research on chemical composition and properties is being carried out under contract at Evans Research and Development Corporation, New York, N. Y., on the isolation, identification and characterization of flavor and aroma components of processed peanut products; at the Agricultural Experiment Station, Oklahoma State University, Stillwater, Oklahoma, on a study of the relation of the carbohydrate, amino acid and protein components of the peanut to the formation of flavor and aroma during roasting; at the Agricultural Experiment Station, Auburn University, Auburn, Alabama, on a study of the limiting environmental conditions for the elaboration of mycotoxins in peanuts; and at the Agricultural Experiment Station, Texas A & M University, College Station, Texas, to develop information relating processing methods, preprocessing history, distribution of immature, mature and germinating peanuts, and external conditions such as mold incidence as they affect consumer-use properties of processed peanut products.

The Federal in-house scientific research effort in this area totals 7.4 professional man-years. All of the present effort is on chemical composition and physical properties. The contract research involves an additional 4.1 man-years, all of the effort being on chemical composition and physical properties.

PROGRAM OF STATE EXPERIMENT STATIONS

State stations have a continuing program of research directed toward increased use of peanuts. A portion of this effort is directed to fundamental studies on fermentation products produced by fungi growing on peanut substrates, including determination of the cultural conditions influencing the production of "toxins" by selected fungi growing on peanuts and peanut oil media. Other compositional research has as its objective the determination of the effects of storage environment and time on chemical, biochemical and physical changes in peanuts and the relationship of these changes to odor, flavor and certain nutritive factors. One study of the quality of peanuts and peanut products has been in progress over ten years. It involves determination of the effects of temperature, time, moisture conditions, storage and packaging on quality (staleness and rancidity development) in peanuts and peanut products. Other research supports the breeding programs through providing data on the susceptibility of various strains of Spanish peanuts to rancidity development.

Studies of the aroma and flavor of peanuts involve consideration of the agronomic and biochemical factors responsible for the flavor of peanuts and peanut butter. Attempts are made to characterize the substances responsible for the aroma and flavor peculiar to roasted peanuts.

Mechanization of peanut harvesting has led to renewed interest in peanut curing studies. Investigations aimed at developing more dependable and efficient methods for curing peanuts of high quality are in progress. Improvements in methods for measuring peanut maturity are sought The effects of various curing treatments are evaluated in terms of flavor,

adherence of the testa to the cotyledons, resistance to splitting and rheological properties of the peanut products. Influence of variety, stage of maturity and curing practices are also considered.

Development of products containing peanuts is pursued in an effort to extend the use of peanut butter, salted peanuts and peanut oil by improvement of present products or through development of new products. Nutritive value and consumer acceptability guide these researches.

The use of peanut oil meal as a source of protein for chicks is also being studied.

The station program on peanut utilization involves approximately 3.4 professional man-years.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Chemical, Physical, and Biological Properties and Structural Factors of the Proteins. The composition, properties, structural factors, and reactions of the proteins and associated materials of various seeds, including peanuts, are being investigated in a program of pioneering research conducted by the Seed Protein Pioneering Research Laboratory. A major objective of this work has been to examine existing methods of classifying seed proteins and to provide the basis for development of new classifications having more meaning in terms of the function of the proteins in seeds. The present system of classifying seed proteins on the basis of solubility and the procedure of naming new proteins by trivial names has long passed its usefulness. Actually the name "seed proteins" is an anachronism that implies a mélange of proteins with many different structures and activities. This kind of a designation was made in the early days of biochemistry when people were beginning to catalog things as they observed them in organs, but as biochemistry progressed and specific activities could be attributed to various constituents, such catalogs disappeared. The one on seed proteins is one of the few remaining, probably because the seed is quiescent.

The proposed classification of seed proteins in storage tissue is based on their location in subcellular particles. Those in the particles have been named aleurins: proteins which occur in protein bodies in seeds, easily identifiable by electron microscopy and separable from other cellular particles or cytoplasmic proteins by suitable techniques. There would be a class of aleurins in Arachis hypogaea, another class in Pisum sativum, and another one in Gossypium hirsutum, etc. This new classification will influence new approaches in protein isolation based on particle separation; in one instance this has resulted in greater fractionation than was accomplished by the classical procedures. It also will speed up study of the biological properties of the major seed proteins and will promote interest in the ultrastructure of seeds. Many of the classical major seed proteins may turn

out to be primarily aleurins. It may turn out that some aleurins will have common functions and structure. These may be common to many seeds, or even to all seeds. Thus far, few proteins have been so handled as to fit into the category of aleurins. Some proteins of the peanut fit into this category; so do proteins from a particular fraction of pea cotyledons, and the acid soluble proteins of the immature wheat kernel.

Protein bodies occur in all storage tissues of seeds which have been investigated. It is the same whether the storage tissue is the cotyledon, as in legumes and cottonseed, or whether it is in the endosperm, as in grains, castor bean, and tung nut. This point was emphasized by a careful study of the ultrastructure of the storage tissue of four oilseeds: peanuts, cottonseed, tung, and castor.

Two subcellular particles were isolated from the cotyledon of the peanut (Arachis hypogaea): one contains 50 to 55% protein and about 10% lipid and and a second, lighter particle, which is stable at pH6, contains almost half and half proteins and lipids. The latter particle also has measurable phosphatase activity. The proteins of these two particles were studied by chromatography on DEAE cellulose. Most of them are predominantly group IV proteins; these are the aleurins of the peanut cotyledon; these are the same as the classical arachin fraction. This work demonstrates another virtue of the particle approach, because it was possible to fractionate the major proteins of the arachin fraction by fractionating the two particles: two fractions which chromatographed differently on DEAE cellulose were obtained.

A careful study was made of the development of protein bodies in the cottonseed (Gossypium hirsutum) at the level of ultrastructure. Two points seem to be clear. First, there is a single membrane around protein bodies in immature cottonseed cotyledons. Other investigators, studying wheat endosperm, have suggested that the protein bodies have a double membrane. This is an important issue in classification. If protein bodies have a single membrane, they are in the same class as vacuoles; if there is a double membrane, they are in the class of plastids. The evidence here would seem to be that they are in a class of vacuoles. Secondly, the proteins seem to be synthesized by extending part of the endoplasmic reticulum in a loose large body surrounded by a single membrane; this body becomes smaller as the seed matures. There is no evidence that the major protein bodies are accumulated by mechanisms similar to those which operate in the pancreas, that is, by the use of the Golgi apparatus.

Important new equipment and techniques have recently been developed to facilitate the study of proteins. Progress has been made in understanding the characteristics of polyacrylamide gel and in transforming it into a sensitive column electrophoresis medium, both for analysis and preparation. Two major operational problems were solved. The first was the stabilization of the gel. Since the gel expands in certain buffers, it is not possible to continue electrophoresis for a long period. It is not clear why the gel expands; it could partly be due to electroendosmosis. It was discovered

empirically that a mixture of tris and phosphate buffers incorporated into the gel medium will stabilize the column; this stabilization is independent of the buffers used in the electrode chambers. The other question was that of yield. Because most of the protein adhered to the cellophane membrane between the column and the anode, yields up to only 30% were obtained at first. This problem was solved by treating electrically charged membrane as if it were permanently charged as, for example, DEAE cellulose, and increasing the salt concentration in the wash. Now yields of 80 to 100% are regularly obtained.

Another useful tool is the study of enzymes by calorimetry. Phosphatase activity is usually measured by taking aliquots during the course of a reaction and determining the amount of inorganic phosphorus released. This reaction cannot be followed continuously. But by taking advantage of the heat of the reaction, it is possible to follow phosphatase reactions by calorimetry from the beginning to the end. This procedure was tested on various phosphatases: potato apyrase, the transport phosphatase of red blood cell ghosts, and a phosphatase from germinating peanut seeds. When the enzyme is acting as a phosphatase without any transport properties, it produces a calorimeter pattern different from that produced when it is acting as a transport phosphatase. Hence, calorimetry has introduced a new dimension in study of enzyme reactions.

Further work on the biochemistry of seeds and their proteins strengthens the notion that seeds are useful models for important biological studies: studies on active centers of enzymes, isolation of cofactors for lipases, changes in ribosomes with state of tissue, and studies in protein synthesis.

It is generally considered that transport of sodium and potassium across membranes is mediated by an adenosinetriphosphatase (ATPase) which is inhibited by the cardiac glycoside, ouabain. All of the work on this enzyme was originally from animal tissues. Such a transport enzyme was isolated in germinating peanut seeds; it responds to changes in sodium, potassium and magnesium concentration; moreover, its activity is affected by ouabain. Here is another instance of the universality of certain basic biological mechanisms and another indication of the possibility of using seeds to study fundamental biological problems.

The castor bean lipase as studied to date is particulate. It was solubilized by incubation at controlled pH, apparently by the action of a proteolytic enzyme contained in the particulate material. It is now completely soluble and can now be purified by standard techniques. The natural cofactor of castor lipase was found to be a cyclic polymer of ricinoleic acid. Other lipid materials which can act as cofactors include ricinoleic acid, oleic acid, monoricinolein, tocopherol succinate, and the methyl half ester of dodecenyl succinate. It is interesting that most compounds with cofactor activity have a free carboxylic acid and a long hydrophobic chain.

There is no information on whether resting seeds contain ribosomes.

Certainly they exist in the actively developing seeds and exist again in the germinating seeds. By taking advantage of work previously done in this laboratory on nonaqueous fractionation of peanuts, a fraction high in nucleic acid was used as a starting material from which were isolated ribosomes, both in cottonseed and peanut cotyledons. The ribosomes from the two seeds are the same; they have a sedimentation value of 80S and are sensitive to magnesium concentration. They appear similar in physical and spectroscopic properties to ribosomes from other tissues. Isolation of ribosomes from the resting seeds provides a good ground state source of ribosomes, that is, a source of ribosomes from tissue in which little, if any, protein synthesis is taking place. It will be interesting to compare the properties of these ribosomes to those in developing and germinating seeds.

In a new line of research, it has been shown that cyclopropene fatty acids inhibit the sulfhydryl group of an SH-sensitive enzyme, castor bean lipase. Lipids containing cyclopropene fatty acids, including certain cottonseed oils and the oil from Sterculia foetida, when fed to hens or to other animals, have been reported to disturb the metabolism of lipids, change the properties of the vitellin membrane, and increase the ratio of stearic to oleic acid in the tissue fat. Castor bean lipase, which had previously been shown at this laboratory to be sensitive to sulfhydryl reagents, was used to test a suggestion in the literature that cyclopropene compounds may react with sulfhydryl compounds to form stable derivatives containing thioether bonds. Sterculia foetida oil inhibited castor lipase, and cottonseed oils containing higher amounts of cyclopropene fatty acids were hydrolyzed at a lower rate than were oils containing a lower quantity, such as refined oils. That the cyclopropene moiety was responsible for these effects was clearly demonstrated when sterculene, a synthetic cyclopropene derivative, was also shown to inhibit the castor lipase. Since these natural oils containing cyclopropene fatty acids have some properties analogous to those of substrates for the enzyme, this work may support the theory that the physiological activity of cyclopropene fatty acids is by way of their reaction with the functional sulfhydryl sites of enzyme systems. It will be interesting to continue studies on the inhibition of a purified soluble castor lipase, which may be a means of labeling the sulfhydryl group responsible for the activity of the enzyme.

2. Identification of Constituents and Factors Influencing Flavor, Aroma, Color, Structure, and Nutritive Value of Processed Products. Further progress has been made in isolating and characterizing minor constituents of peanuts and in establishing their physiological activity. Three additional products have been isolated from defatted peanut flour. A new amino acid, N-methylhydroxyproline, was isolated and characterized--the first report of this amino acid as a constituent of an edible product. It is probably a methyl donor; if so, it could play a significant role when peanuts are used as food. Also isolated from the flour was pipecolic acid, which is also of potential importance since it is probably involved in the metabolism of lysine. A peanut factor is known to play a role in reducing bleeding time, especially in hemophiliacs. Another constituent isolated, apparently a

purine, causes marked relaxation of excised smooth muscle. This myotonic factor in peanuts -- which may be related to the hemostatic factor -- apparently is a coenzyme involved in the release of energy in the transformation of adenosine triphosphate to adenosine diphosphate, not only in muscle contraction but also in the formation of fibrin from fibrinogen and in the agglutination of the blood platelets.

Because of their potentially marked physiological activity, these minor constituents may be of considerable significance and importance to the role of peanuts in the human diet. This research project has been terminated and available personnel are being utilized to augment research on fungi and toxic fungal metabolites in peanuts. (S4 1-100).

In an investigation complementary to the preceding work, basic research on the nonglyceride lipid-soluble constituents of peanuts has been continued. Solvent fractionation and chromatographic separation of nonglyceridic components from the bulk of the triglycerides was investigated. Column chromatographic procedures proved feasible for separating crude commercial peanut oil into a series of simple mixtures but have not yet been successful for the preparation of pure compounds. Partition of the crude oil between hexane and acetonitrile was found to result in considerable enrichment of three or four of the nonglyceride components in the acetonitrile phase. Emphasis will be placed on the components in the acetonitrile extract. By a combination of solvent partition and column and/or thin layer chromatography, it should be possible to obtain sufficient amounts of lipid-soluble peanut constituents for instrumental examination. Volatile fractions will be investigated by gas chromatography. (S4 1-109).

The contractor (Evans Research and Development Corporation) is conducting research on the isolation, identification, and characterization of flavor and aroma components of processed peanut products to form the basis for producing improved peanut products of greater consumer acceptability. Preliminary experiments revealed that steam distillation at atmospheric pressure was not satisfactory for concentrating a "true" peanut aroma. Hexane extraction, however, yielded a concentrate rich in peanut aroma.

Modification of the solvent extraction procedure achieved convenient extraction of large quantities of peanuts and rendered subsequent fractionations more complete. High grade, medium roasted peanuts (Spanish, including southwestern-grown, and either Virginia or Runner type) are being used in the research. Thus far, two aldehydes -- 2,4-decadienal and hexanal -- have been identified in a neutral oil fraction, and acetic and isovaleric acids in an acid fraction. A third acid is either propionic or isobutyric acid, and a fourth one is probably a branched-chain, seven carbon acid. The identification work is continuing. Investigation of flavor precursors in raw peanuts also is in progress. (S4 1-106(C)).

3. Investigation of Occurrence, Determination, and Properties of Fungi and Toxic Fungal Metabolites Which may Develop in Peanuts and Their Processed

Products. The problem of agricultural commidities' becoming contaminated with toxic microbial metabolites is receiving increasing recognition. To determine the prevalence of such contamination in peanuts, two surveys based on chemical assay are currently being conducted. For the first test, the Biometrics Service of ARS designed a sampling plan in which a cross section of the 1963 crop from warehouses storing CCC's stock was analyzed by Southern Division scientists using their own new technique, which is sensitive to an aflatoxin content of 2-3 parts per billion. Sampling included 5 states, 3 types of peanuts, and 4 classifications; results from 137 samples indicate correlation between quality classification and presence and degree of contamination. The data obtained are of particular and immediate value, in that they supply good leads toward methods for eliminating contaminated peanuts from food channels and furnish a basis for the development of an orderly marketing program for the 1964 peanut crop. The second test, on stocks of No. 2 peanuts, is presently being performed in cooperation with other groups, all of which use the same sampling and analytical procedures sensitive to 50 parts per billion, the limit of sensitivity of present bioassay procedures.

In addition to the two chemical assays, extensive animal feeding tests are being conducted: aflatoxin-free meals, meals containing aflatoxin, and a mixed solvent extract of an aflatoxin-containing meal have been sent to the Pharmacology Laboratory, WU, to determine the effect of aflatoxin in peanut meal rations.

The method of extracting aflatoxin developed by Southern Division scientists employs a solvent mixture composed of acetone, hexane, and water. The same mixture has also been found to remove aflatoxin from contaminated peanut meal. Since aflatoxin can readily be removed from oil by conventional refining procedures, this mixture may become the solvent of choice in commercial processing of peanuts to oil and aflatoxin-free meal. (S4 1-116).

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AREA NO. 8 - TUNG PROCESSING AND PRODUCTS

Problem. Tung oil has lost much of its traditional market in protective coatings to synthetic raw materials. New and improved industrial products from tung oil must be developed to recapture lost markets, maintain present markets, and provide new outlets for surplus tung oil. Basic information is needed on the chemical composition and properties of tung oil and its fatty acids, and on the chemical modification of these materials to permit more effective exploitation of their unique characteristics in protective coatings, agricultural and industrial chemicals, surfactants, and plasticizers. For example, improved coatings utilizing tung oil are needed to meet increased performance demands and competition from synthetic polymeric coatings. Intumescing fire-retardant coatings and water-reducible coatings containing tung oil are desired. A limited market of low economic value exists for tung meal as a fertilizer. Research is needed to develop more information on profitable uses of tung meal to benefit the overall economy of the tung industry.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving organic chemists engaged in both basic and applied research on tung and its products. Emphasis in the present program is on development of new and improved industrial products from tung oil and its derivatives.

Research is conducted at New Orleans, Louisiana, to develop fundamental information on the chemical composition, properties, structural factors, and reactions of oilseed proteins, as a basis for development of new concepts and possibly new uses for oilseed proteins.

Research to develop new and improved industrial products from tung oil is carried out at New Orleans, Louisiana, with cooperation and support by the Pan American Tung Research and Development League and the U. S. Army Engineer Research and Development Laboratories. The League maintains a part-time Fellow for research on the production of improved protective coatings. Major emphasis is placed on the development of exterior and interior, intumescing, fire-retardant surface coatings from tung oil and tung oil derivatives. Tung alkyds are being chemically altered and formulations modified to produce coatings which will intumesce to give a thick cellular, fire-resistant material upon thermal or flame exposure. The U. S. Army Engineers evaluate the more promising fire-retardant coating formulations developed with their support.

Other research in the area of chemical composition and physical properties is in progress under a grant of P.L. 480 funds to the National Chemical Laboratory, Poona, India, for investigations of the effect of heat on tung oil and its derivatives, and characterization and identification of compounds

resulting from heat treatments, to extend the utilization of tung oil (project duration - 5 yrs.).

The Federal in-house scientific research effort in this area totals 4.9 professional man-years. Of this total 0.7 is devoted to chemical composition and physical properties, and 4.2 to new and improved industrial products. P.L. 480 research involves 1 grant for research on chemical composition and physical properties.

The following line of work was terminated during the year: (1) Chemical modification of tung oil and its fatty acids to produce materials having potential industrial utility (under new and improved industrial products).

PROGRAM OF STATE EXPERIMENT STATIONS

State stations did not report work in this area.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Structural Factors, Properties, and Reactions of the Protein. The composition, structural factors, properties, and reactions of oilseed proteins and associated materials are being investigated in research conducted by the Seed Protein Pioneering Research Laboratory. The basic information developed should lead to new concepts and possibly new applications for oilseed proteins, including tung protein. Since peanuts were found to be a particularly suitable experimental material and were employed for much of the early pioneering research on seed proteins, the report of progress in this research is given in Area No. 7, "Peanuts Processing and Products," as in the previous report.

2. Basic Investigations of the Effect of Heat on Tung Oil and Its Derivatives. At the National Chemical Laboratory in Poona, India, research to investigate the effect of heat on tung oil and on its derivatives has been initiated under a P.L. 480 grant. Compounds resulting from subjecting these compounds to heat will be identified and characterized. The early stages of the work have been confined to methyl esters of alpha- and beta-eleostearic acid. A chromatographically pure cyclic monomer has been isolated from methyl alpha-eleostearate thermally treated in an evacuated sealed tube. The fundamental information generated by this research is expected to aid in the development of new industrial uses for tung oil outside the protective coatings field. (UR-A7-(40)-12).

B. New and Improved Industrial Products

1. Intumescing Fire-Retardant Surface Coatings from Tung Oil Alkyds. Considerable progress has been made in the development of water-resistant, intumescing, fire-retardant coatings based on tung oil and its derivatives,

research conducted with the cooperation and support of the U. S. Army Engineer Research and Development Laboratories and of the Pan American Tung Research and Development League. Use of the elementary 8-foot tunnel furnace devised at the Southern Division to screen experimental coatings has provided insight into the reasons that an early formulation which performed well in the standard fire test cabinet did not do so in the Underwriters' Laboratories' 25-foot tunnel furnace. From these observations, experimental formulations have been developed to eliminate the excessive blistering, rupturing, and peeling of the earlier coatings. The newer coatings, formulated from vehicles based on chemically-modified tung oil and from chemically modified, water-resistant carbonific and spumific components, intumesce under heat to produce an insulating layer of carbonaceous material. The two formulations that performed best in the 8-foot tunnel furnace also gave encouraging results in the 25-foot tunnel furnace: one had a respectable flame spread value of 40; the other, between 45 and 55 -- significantly better than the value of 85 exhibited by earlier coatings tested in the UL furnace. As far as known, there are no practical commercial, water-resistant, fire-retardant coatings available with such low flame-spread values. However, attempts will be made to further improve the new formulations, including lowering their flame-spread values to 25 if possible. There has been considerable industrial interest in such coatings, which have potential for commercial, civilian, and military use. (S4 1-113).

2. Chemical Modification of Tung Oil to Produce New and Improved Products, Such as Protective Coatings, Agricultural Chemicals, Surfactants, and Plasticizers. Several approaches for chemical modification of tung oil and its fatty acids to produce materials having industrial utility were investigated. In further research on reactions to improve the surfactant or emulsifier properties of tung oil products for use as fugitive emulsifiers, difficulty was encountered in attempts to prepare the nitrile from eleostearic acid by reaction with ammonia. The carboxyl group was modified only to the amide stage, and eleostearate unsaturation was reduced considerably. However, recent experiments have resulted in the successful preparation of ethenoxylated monoglycerides of tung oil with retention of the conjugated triene unsaturation of the eleostearate moiety. This result could lead to improved fugitive emulsifier products from tung oil. In the reaction employed, tung monoglycerides are reacted with ethylene oxide using bis(2-methoxyethyl) ether as solvent, catalytic amounts of metallic sodium, temperatures close to 150° C., pressures of 1/2 to 1 atmosphere above atmospheric, and short reaction times. The products are mixtures of materials containing on the average 1-4 moles of ethylene oxide per mole of tung monoglycerides, the average amount of ethylene oxide reacted depending on the pressure and length of the reaction time. (S4 1-93).

PUBLICATIONS -- USDA AND COOPERATIVE PROGRAMS

New and Improved Industrial Products

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1/ Publication resulting from research supported in part by funds transferred from the U. S. Army Engineer Research and Development Laboratories.

AREA NO. 9 - CITRUS AND SUBTROPICAL FRUITS PROCESSING AND PRODUCTS - SOUTHERN LABORATORY

Problem. The citrus and subtropical fruit production of the Southern Region is an expanding industry with the need for the development of better, as well as new-type consumer products, and for the improvement of present or invention of new processing procedures and machinery. These advances are required to regularly utilize the currently large production, particularly of oranges and grapefruit, and the anticipated higher production of these fruits, to the economic advantage of the growers and consumers. Basic research is needed to lay the groundwork for these advances. This research is needed, for example, on the composition and physical nature of essential oils, flavonoids, including bitter constituents, constituents responsible for oxidized off-flavors, carotenoids, and the like, which determine many of the sensory characteristics, and which affect product quality and stability. Other problems whose solutions are dependent upon the availability of more detailed compositional and physical data are: cloud stability, gelation, discoloration, fermentation, and the like. Increased production of citrus has stimulated the development of new products but many of these are urgently in need of improvement which will depend in part upon advances in basic research. New products are needed to attract new markets and also to reduce packaging and shipping costs. Research is needed to improve frozen citrus concentrates as processing procedures change, to develop better high density concentrate products, citrus powders, chilled juice and section products, pulp-fortified products, and to develop new or improved canned products which have a natural fruit flavor. Research is especially needed on grapefruit to develop practical methods for reducing the bitterness and harshness of juice products and to increase the use of grapefruit juice base in mixed fruit juice blends, drinks, concentrates and the like. Along with progress on product development there is a serious need to improve the actual processing procedures, processing equipment, and packaging operations and materials, to obtain and maintain the most desirable fruit characteristics. As an illustration, research is needed to develop less expensive dehydration equipment and an improved process for the production of citrus powders.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving biochemists, organic chemists, food technologists, and a chemical engineer engaged in both basic and applied utilization research studies on citrus and subtropical fruits of the Southern Region to develop new or extended uses for these commodities.

Research to develop basic information on chemical composition and physical properties of citrus and subtropical fruits, and their products and by-products is conducted at the U. S. Fruit and Vegetable Products Laboratories

at Weslaco, Texas and Winter Haven, Florida. This information provides the necessary basis for efficient research in developing new and improved food products and processing technology. At the Weslaco Laboratory the program includes investigations of the biochemical mechanism of the conversion of precursors to carotenoids in grapefruit as a basis for improvement of processing characteristics of and products from colored grapefruit. The Texas Agricultural Experiment Station (substation 15, Weslaco), Citrus Rootstock Investigations Laboratory (CR, ARS, Weslaco), and the Texas College of Arts and Industries are providing grapefruit of known history and conducting, or cooperating in conducting, on-the-tree tests. Additional research on chemical composition and physical properties is carried out under contract at the University of Oklahoma Research Institute, Norman, Oklahoma, on investigations of the effect of maturity of grapefruit on total flavonoids, naringin, and poncirin; and on the chemistry and nature of naringin and naringin-derived compounds to provide a scientific basis for the control of bitterness in processed grapefruit products. At the Winter Haven Laboratory the program includes research to identify recently isolated flavones and other neutral orange peel constituents and evaluate their relation to bitterness and harshness in orange products. Investigations are also in progress on the composition of essential oils in citrus products particularly orange, to provide a basis for improvement in quality and uniformity of citrus products; on investigations of the chemical and physical nature of components of cloud of orange juice to provide better understanding and control of factors affecting stability of orange juice products; and on investigations of the identities, quantities and chemistry of components in Florida grapefruit responsible for excessive bitterness and harshness in processed products. Close consultation is maintained with the Florida Agricultural Experiment Station (Citrus Experiment Station, Lake Alfred) and the industry.

Research to develop new and improved food products is carried out at the U. S. Fruit and Vegetable Products Laboratories at Weslaco, Texas, and Winter Haven, Florida. At the Weslaco Laboratory the major applied effort is to develop products which will make greater and more efficient use of grapefruit. Emphasis at the present time is on the utilization of natural and debittered grapefruit juice and puree as bases for the development of improved fruit juice blends, drinks, and concentrates. This research is being carried out in part in cooperation with several state and private organizations. The cooperators provide fruit or raw materials, such as pulp and juice, of known history. Processing plant facilities are available from the Texsun Citrus Corporation (Weslaco) and Rio-Vac, Inc. (Harlingen). Formal agreements exist with the Texas Agricultural Experiment Station (College Station and Weslaco), with Texsun Citrus Corporation (Weslaco) and with Rio Farms, Inc. (Edcouch). Informal cooperation is maintained with Texas Citrus Mutual, Inc. (Weslaco), Texas Canners Association (Weslaco) and such other organizations as are found necessary for the procurement and processing of fruit. At the Winter Haven Laboratory research is in progress to develop high quality, "instant" citrus powders by new and improved processing technology as described below.

In the field of new and improved processing technology, research is being carried out at the U. S. Fruit and Vegetable Products Laboratory, Winter Haven, Florida, to determine how the "foam-mat" type of air-drying can be applied for the preparation of dried citrus products of optimum flavor and stability. Foam-mat drying of orange juices, and grapefruit juices, is currently being studied. This research is conducted in cooperation with the Western Utilization Research and Development Division (ARS) and the Florida Citrus Commission under a formal memorandum of understanding. Additional research on new and improved processing technology will be carried out under contract at the Citrus Experiment Station, University of Florida, Lake Alfred, Florida, on the development of a practical and efficient pilot plant scale process for the production of enzymatically debittered grapefruit juice and products with improved flavor, product stability and storage characteristics.

The Federal in-house scientific effort at the Southern Division devoted to research in this area totals 20.9 professional man-years. Of this total 12.8 is devoted to chemical composition and physical properties, 2.8 to new and improved food products, and 5.3 to new and improved processing technology. The contract reseach involves an additional 1.9 man-years, 1.4 being on chemical composition and physical properties, and 0.5 being on new and improved processing technology.

PROGRAM OF STATE EXPERIMENT STATION

The States are engaged in research on the utilization of citrus and subtropical fruits in an effort to expand markets through increased use. Research on citrus begins with efforts to reduce decay during storage and transit through control of the physical, biochemical and physiological changes in citrus during handling--i.e., the effects of precooling and study of factors of temperature, humidity and air flow during cooling.

New product studies involve determination of the basic product characteristics, flavors, types and components and relate these to market demands. A thorough examination of the sources of flavor of some common foods including citrus is in progress. A phase of this program deals with the effects of oxidation and/or hydration on the flavor and aroma of the terpenes of citrus and the role structure plays in odor production. Conversion of citrus terpenes to useful chemical compounds is also under study. Another study has as its goal to extract, separate, identify and determine quantitatively each of the volatile components responsible for the natural flavors and occasional off-flavors in citrus fruits, citrus oils and processed citrus products.

The characteristics of commercial frozen Florida orange concentrate and superconcentrate are frequently determined to establish physical and chemical characteristics of the products. Base juices are prepared from citrus fruits and used to determine the effects of the fruit components on the characteristics of frozen citrus concentrate.

Firming of canned grapefruit sections with calcium salts and other materials is also under study. Characteristics of canned and concentrated juices are determined initially and after storage at elevated temperatures.

Utilization of citrus waste is receiving attention. In one study, isolated cultures from natural sources are being used to investigate production of glycerol and glycols from citrus wastes by fermentation.

The program with other subtropical crops such as guava, mango, soursap, banana, pineapple, coffee, and plantain includes production of freezedried products of high quality and good storage life. The economic feasibility of expanding markets for subtropical agricultural products through processing and utilization of new products is being further tested by preparation of soft drinks from tropical fruits--carbonated and noncarbonated, canned and bottled. Basic biochemical and microbiological studies of tropical foods are directed to discovery of special methods, special properties or nutritional qualities which may be used in new product development. Production of such products as banana puree, fried snack items, flakes, flours, fruit powders, flavoring extracts, candied items, canning syrups, nectars and juices is under study.

In addition, attempts to isolate the enzymes of fig latex are underway. The isolated proteolytic enzymes are characterized as to molecular weight, activity and amino acid composition.

Two stations, Hawaii and Puerto Rico have programs designed to improve the economic condition of their coffee industries. These researches embrace work on the microbiology of the coffee fermentation process, on the drying of coffee, and on the quality and acceptability of the final product.

The total research effort in citrus and subtropical fruit is about 21.6 professional man-years.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Chemical and Physical Properties of Flavoring Constituents of Florida Citrus and Subtropical Fruit Products. In basic work on the composition of essential citrus oils, a new terpene (2,4-p-menthadiene) and two new sesquiterpenes (valencene and β-copaene) have been isolated from orange oil. β-copaene has also been isolated from grapefruit oil. The sesquiterpenes copaene, α-ylangene, β-elemene, α-and β-humulene, and delta-cadinene have been shown to be constituents of both orange and grapefruit oil. β-ylangene and valencene are two of the major sesquiterpenes in orange oil; valencene, the major sesquiterpene in orange juice; and β-caryophyllene, the major sesquiterpene in grapefruit oil. The structure of both α- and β-ylangene has been determined, the former being stereoisomeric with copaene. Valencene possesses nootkatone structure, whereas β-copaene, except for having

an exocyllic terminal double bond, is identical to α-copaene. Research progress has been aided by development of a thorough, rapid method for determination of terpenes and sesquiterpenes in citrus oils by use of the combined Time-of-Flight Mass Spectrometer and capillary gas chromatograph. As a continuation of the present project, composition studies will be pursued under a new related project. The research will include elucidation of structure of flavor-contributing compounds and particular emphasis will be placed on alcohols and compounds of low molecular weight (high volatility). (S3 2-36).

2. Investigation of Bitter Principle and Flavonoids in Citrus Products. A thin-layer chromatographic system has been devised to permit separation and form the basis for quantitative determination of the five flavones thus far encountered in the neutral bitter fraction of orange peel juice extract. Included among these flavones are tangeretin, heptamethoxyflavone, nobiletin, sinensetin and new flavone. Preliminary work on the new unidentified flavone suggests that it is a tetramethoxyflavone, perhaps the 5,6,7,4' compounds. In some samples, there was evidence of a second unknown constituent, which has not as yet been isolated. In contrast to prevailing thought, orange peel juice produced late in the 1962-63 season appeared to have increased bitterness compared with that produced earlier; yield was also decreased. However, because of the severe freeze in December, 1962, these data may be atypical. Initial monitoring tests on orange peel juice for the 1963-64 season revealed a slightly lower taste threshold, a slightly higher benzene extract content, and an apparently greater contribution of the neutral fraction to total peel juice flavor than those observed for corresponding periods of the preceding season. If these trends are maintained throughout the season, the information will be of practical importance in commercial extraction operations. Research will be continued under a new project to identify recently isolated flavones and other neutral orange peel constituents and to evaluate their relation to bitterness and harshness in orange products. (S3 2-37).

The University of Oklahoma Research Institute is conducting contract research to investigate the changes that occur as grapefruit matures. Efforts are continuing to develop a quantitative analytical procedure for the determination of flavanones, including bitter constituents, in grapefruit juice and products. The procedure under investigation involves quantitatively isolating the flavanones, separating the individual flavanones (naringin, poncirin, prunin, and naringenin) by paper chromatography, and measuring their fluorescence intensities. A newly formulated benzene-acetic acid-water-nitromethane solvent has given good results when employed in the paper chromatography. Polyamid or Polyclar AT columns appear to be promising tools for separating many of the constituents of grapefruit juice. Pure poncirin for use as a standard has been prepared from extracts of trifoliate oranges; and early and midseason Ruby Red grapefruit (1963-64 season) have been frozen for future use. Major effort will continue on quantitatively separating and determining the individual flavanones. (S3 2-39 (C) (Rev.)).

A new project has recently been initiated for investigation of the identities, quantities, and chemistry of components in Florida grapefruit.

responsible for excessive bitterness and harshness in processed products. Following exploratory experiments on a laboratory scale, pilot plant extraction of Florida grapefruit was begun, but difficulties encountered with the process led to resumption of work on a laboratory scale. Approximately 45 pounds of grapefruit have been extracted and the volumes reduced to a workable size for isolation and identification. Column chromatographic procedures, thin-layer chromatography, various colorimetric tests, and spectrophotometric procedures have been investigated for use in the isolation and identification of flavonoids in grapefruit extracts. (S3 2-42).

3. Factors Affecting the Physical Characteristics of Processed Citrus Products. Much of the flavor of citrus is due to incorporation of peel oil into the juice, evidently--as shown by recent work--by its being dissolved in suspended lipids. This finding may explain the observations that the flavor of the juice changes upon standing and that an increase of suspended solids, within limits, improves its flavor. Examination of "cloud" fractions from additional samples of orange juice confirmed the previous conclusion that juice from freeze-damaged fruit contained a higher percentage of solvent-insoluble solids than did normal juice. Because a severe freeze rendered material collected from oranges during the 1962-63 season atypical, additional samples of cloud were collected from early and midseason oranges of the 1963-64 season. Analyses for lipids, carbohydrates, ash, and nitrogen have not revealed important differences associated with season or variety during the past two years. However, differences between pulp and fine cloud and between suspensions prepared from different parts of the fruit indicate that source of cloud is important. The natural cloud (very fine particles), which contains much of the orange flavor, appears to come from the juice rather than from mechanical disintegration of structural tissue. Attempts to separate cloud components by gel filtration or by application of electric potential to suspended cloud proved ineffective. Plans have been made for collection, processing, and analysis of samples of juice, pulp washings, and centrifuge sludge during the Valencia season for comparison with previous seasons' samples. The project for investigation of the chemical and physical nature of components of cloud has been revised, based on research results to date. (S3 2-38)(Rev.)).

4. Basic Investigations of Carotenoids in Grapefruit. Fundamental studies of the nature and mechanism of the biosynthesis of carotenoids support the theory of parallel synthesis of the carotenes. Data obtained from labeling tomatoes with $C^{14}O_2$ and 2-C^{14}-mevalonic acid indicate that although phytoene may be a precursor for other carotenes, including lycopene, it is probably not a pool precursor. As an extension of this basic work, the effects of the environmental factors of temperature and light on the pigments of red grapefruit are being evaluated. On-the-tree experiments with red grapefruit showed that cold temperatures (60° F days, 40° F nights) caused degreening of the fruit, cessation of growth, and a decline in lycopene content, whereas fruit at high temperatures (95° F days, 85° F nights) remained green, continued to grow, and retained high lycopene content despite rapid sizing. When grapefruit attached to a tree were exposed to labeled carbon dioxide,

the amount of label in the carpels 48 hours after exposure was about the same whether the fruit was exposed in the light or in the dark. However, label in the peel was about 30-50 times higher after light exposure than after dark exposure, since in the light very active photosynthesis fixes CO_2 before it diffuses through the peel. Photosynthesis and photosynthetic efficiency (CO_2 fixed per mg. chlorophyll) reach a minimum approximately when lycopene concentration reaches a maximum. The carpels then appear capable of total synthesis of carotenes; there is little probability of precursors from leaves or stems playing a major role in carotene synthesis within the fruit. The evidence that temperature is a major factor is the first indication that anything outside the fruit can influence the concentration of lycopene on the inside. Such information, together with the biochemical data, may introduce a new theory of biosynthesis, determine optimum harvesting time, and ultimately permit control of color in the fruit. Future plans include an investigation of noncarotenoid lipids having chromatographic behavior similar to that of the carotenes. (S3 2-34 (Rev.)).

B. New and Improved Food Products

1. Development of New Grapefruit Based Beverages. Research is continuing on the utilization of natural and debittered grapefruit juice in improved juice drinks. Preliminary work on resin debittering by use of nylon, Polyamid, or Polyclar AT powders appears promising. It has been shown that resin debittering does not materially change the flavor or composition of the juice, other than to remove bitter constituents, and does not adversely affect the vitamin C content. Resins having greater specific absorption capacities for flavanones are being sought; if they can be found, resin debittering offers an alternative process to use of enzymes. Work to develop accurate tests for bitterness in grapefruit juice and blended drinks will be aided by recent progress on purification of needed flavanones. Completed flavor and color evaluations of grapefruit-strawberry drink concentrates stored 12 months in plain and enamel cans showed that samples in plain tin cans retained acceptable quality for seven months at 68° F, or lower, but frozen storage is necessary if maximum flavor and color are to be retained for as long as a year. The drink is now ready for consumer acceptance testing. Future plans are to purify the flavanones necessary for the development of better tests for bitterness, to evaluate other resins, to study the development and storage of flavonoids in grapefruit before maturity, and to evaluate other blends of grapefruit with fruit or berries. (S3 2-40).

C. New and Improved Processing Technology

1. Application of Foam-mat Drying to Florida Citrus. Investigations of factors affecting processing and product characteristics of foam-mat dried orange juice powder were continued in cooperation with WU and the Florida Citrus Commission. Foam having small and uniform bubble sizes has been formulated to permit reduction of moisture to less than 1% in the final powder without secondary desiccation processing. Achieving this lower

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initial moisture content should make the process more economical, eliminate the need for an in-packing desiccant, and impart a higher degree of storage stability. The most useful release agents and best temperatures and pressures for the "warm-rolling" or "densitizing" treatment have been developed for increasing bulk density of orange powders without damaging organoleptic properties. The "warm-rolling" technique and packaging treatments including vacuum equilibration and use of CO_2 have resulted in improvements in reconstitution and appearance of the juice. In studies of the effect of brix/acid ratio on drying of orange powders, a definite taste preference was shown for powders made from concentrates with brix/acid ratio of 14/1. The addition of antioxidants to orange powders did not improve their storage life at 85° F. Thin-layer chromatography and column chromatography have indicated some relationship between certain carotenoid fractions and the development of off-flavors upon prolonged storage of orange powders at elevated temperatures. These analytical methods may lead to objective measures of storage stability, storage history, or quality. Plans include installation and testing of a crater-type foam-mat dryer, further improvement of reconstitution ease and appearance, determination of the relationship between conditions under which a concentrate is prepared and its drying characteristics, and comparison of freeze-dried with foam-mat dried powders. (S3 2-32; S3 2-43).

Storage tests on grapefruit powders prepared during time/temperature drying studies have been carried out in a continuation of the cooperative study conducted by SU, WU, and the Florida Citrus Commission. Stored as long as 12 weeks at 85° F and more than 16 weeks at 70° F the grapefruit powders have shown no significant change in flavor. Use of new foam formulations has reduced moisture content to less than 1% without employing drying temperatures greater than 180° F or a secondary drying process. This advance obviates the need for an in-package desiccant. In conjunction with the "warm-rolling" or densitizing treatment, recently developed CO_2 packaging treatments facilitate reconstitution and improve appearance of the reconstituted product, eliminating or retarding the appearance of the previously troublesome milky or foamy character. Several grapefruit powder samples have been evaluated for suitability for use in consumer acceptance tests in Western Germany. Future work will involve testing variables related to improved reconstitution and appearance and to stability on storage. (S3 2-41).

PUBLICATIONS -- USDA AND COOPERATIVE PROGRAMS

Chemical Composition and Physical Properties

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AREA NO. 10 - VEGETABLES PROCESSING AND PRODUCTS - SOUTHERN LABORATORY

Problem. Although extensive progress has been made in recent years in developing stable, attractive, and convenient to use vegetable products, new and improved processed products must be developed and means of stabilizing perishable vegetables provided to minimize the adverse effects of seasonable surpluses and unfavorable markets, and to provide an adequate supply of good food for a growing population. Product quality needs to be improved and processing cost reduced through the adaptation and application of the latest technological developments and nutritional findings. For example, a major problem of the cucumber industry, since most of the crop is brine-cured, is to improve the curing process so that no loss occurs in the value of the cucumber during the brine-curing and storage process and the cost of processing is reduced. As another example, a precooked, dehydrated, sweetpotato product has been developed which has good shelf life, when sealed under an inert gas. It reconstitutes to a product having the characteristics of freshly cooked and pureed sweetpotatoes. Applied research on a pilot-plant scale is needed to obtain additional engineering and processing data applicable to commercial production of flakes from different varieties. Basic research is needed to improve the quality and storageability of the product. There is a continuing need in the use of vegetables for processing to investigate the characteristics of the raw material as these characteristics are affected by climate, soil, cultural practices, breeding and the like. Celery, already an important flavoring ingredient, could become much more important if the factors and constituents responsible for the intensity, variableness, and stability of its flavor could be controlled in processing, and processed products of improved flavor and convenience could be developed. Many vegetables grown in the Southern Region differ in their chemical and physical characteristics from the same crops grown in the more temperate regions; and several vegetable crops are grown almost exclusively in the Southern Region. More utilization research is needed to complement the Federal and State production research programs and to provide cooperation in the form of composition and processing studies.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving biochemists, organic chemists, microbiologists, food technologists, and chemical engineers engaged in both basic and applied utilization research studies on vegetables of the Southern Region to develop new or extended uses for these commodities.

Research to develop basic information on chemical composition and physical properties of vegetables, their products and byproducts, is conducted as a basis for efficient research in developing new and improved food products and processing technology. Emphasis at the present time is on investigations

of the flavor and aroma components in natural and pure culture fermented cucumber pickle products, carried out at the U. S. Food Fermentation Laboratory, Raleigh, North Carolina, to provide the basis for producing pickle products of greater consumer acceptability. The North Carolina and Michigan Agricultural Experiment Stations, and the Pickle Packers International, Inc., cooperate in this research.

In the field of new and improved food products by processing of vegetables, both basic and applied research are being carried out at New Orleans, Louisiana, to improve the stability of the flavor of precooked, dehydrated sweetpotato flakes packaged in air, and to improve the processability of uncured sweetpotatoes and their flake characteristics. These are two major problems still facing the new sweetpotato flake industry. Current research approaches involve evaluation of antioxidants and other additives for flavor stabilization, and investigation of enzymes in accelerating the curing of sweetpotatoes. Close cooperation is maintained with the Louisiana Agricultural Experiment Station, and industry and industry associations. Research is in progress at the U. S. Fruit and Vegetable Products Laboratory, Winter Haven, Florida, on the development of processed celery products of improved flavor and convenience. Research is also being conducted at the U. S. Fruit and Vegetable Products Laboratory, Weslaco, Texas, to develop new and improved processed products from southern grown vegetables other than sweetpotatoes and celery. The Texas Agricultural Experiment Station and industry associations provide raw materials of known history for this research.

Research on new and improved processing technology is conducted at New Orleans, Louisiana and at the U. S. Food Fermentation Laboratory, Raleigh, North Carolina. Pilot-plant investigations are being carried out at New Orleans to develop new and improved processing methods applicable to commercial manufacture of stable, precooked, dehydrated sweetpotato flakes from sweetpotatoes of different varieties and environmental history. Processing variables currently being investigated include the effect of variety, curing, type of cooking, drying conditions, and various food additives. Cooperation is maintained with the Marketing Economics Division, ERS, for the market evaluation of improved flake products, and with the Louisiana Agricultural Experiment Station, the Louisiana Sweetpotato Association, the Louisiana Sweetpotato Commission, and various industrial concerns. At Raleigh the objective of the research is to improve cucumber processing technology and the quality of the products. Current emphasis is on investigations of methods for the controlled fermentation of cucumbers by application of pure culture techniques to fermentation practices (including differential control of microbial species in natural fermentations by chemical and physical means) in order to reduce processing costs and improve product characteristics. A hydrolytic enzyme inhibitor extracted from sericea forage which has been under investigation for the prevention of softening of cucumber brinestock during the curing process is now being isolated in quantity for investigation of its chemical properties. Cooperation is maintained with the North Carolina Agricultural Experiment Station. The Michigan State University (Department of Microbiology) is also cooperating by providing technical

assistance in the controlled fermentation studies. The Pickle Packers International, Inc. contributes support to the research and supplies raw material.

The Federal in-house scientific effort at the Southern Division devoted to research in this area totals 19.6 professional man-years. Of this total 2.2 is devoted to chemical composition and physical properties, 10.9 to new and improved food products, and 6.5 to new and improved processing technology.

PROGRAM OF STATE EXPERIMENT STATIONS

State stations have a continuing long-term research effort devoted to vegetable processing and products. Research on the adaptability and evaluation of vegetable varieties for processing is a standard service to plant breeding programs. Before introduction of new varieties, processing yields and processing characteristics are determined. This type of research extends to consideration of the effects of various production variables on processed product quality. Effects of maturity at harvest, mechanical harvesting, fresh product characteristics, post-harvesting handling and storage are examples of problems under study. The degree of correlation or association between color and flavor in fresh and in processed items is always of major concern.

Chemical composition and physical properties are also related to product acceptance and quality. This research ranges from standard composition studies to basic studies of special components. For example, the non-cellulosic constituents of plant cell walls are being investigated. The nitrogen compounds in mushrooms are determined and evaluated as a function of previous history of growth, handling and processing of mushrooms. A continuing analysis of the biochemical changes that occur in vegetables at the different stages of maturity is made. Other studies deal with determination of the antioxidant properties of chile and the antioxidant effects exerted in the various kinds of meats. Estimates of the metal complexes of chlorophyll derivatives in processed foods and their effect on the color of processed foods are made through processing experiments conducted under controlled conditions of metal contamination in the laboratory or in commercial processing plants. The role of enzymes in chemical and physical changes in processed foods is studied through use of purified enzyme systems, substrates and reaction products.

In order to obtain a better understanding of the reasons for changes in flavors during processing and storage and for the development of off-flavors, a comprehensive program on flavors in processed foods is in progress. Heat-induced flavors; lipids in flavor, bitter flavor of carrots, and natural fresh flavors of vegetables are all under study.

Microbiological research extends from study of the natural flora found on fresh vegetables to studies of contaminants found in commercially processed

foods. Methods for microbiological examination of foods are being developed. Physiological, morphological, and nutritional variation among important organisms are determined to facilitate control of the organism or essential understanding of the role of the organism in desirable or useful applications. Bacterial endospores receive much study. It is hoped that this work will lead to the improvement of present methods of sterilization and food preservation. The radioresistance of bacterial endospores and use of combined antibiotics and heat are carefully researched to provide information of use in developing new and improved procedures for canning vegetables. Food poisoning organisms are the object of continuing interest. The incidence of spoilage organisms, survival patterns, and means of control are being investigated. Studies on the effect of carbon dioxide inhibition of microbial growth are in progress. The microbiology of processed foods, for example--dehydrated foods, is another area of research activity.

New and improved vegetable processing technology is sought in studies of freeze-drying, brining, canning, dehydration, fermentation, hydro-cooling and controlled atmosphere methods. Basic studies deal with new techniques of soaking and preparation, enzyme inactivation and regeneration, fluid flow and heat transfer problems. Special attention is being given to development of high-temperature, short-time methods and the advantages of low-temperature handling of sterilized foods. A comprehensive study of the effects of controlled or modified atmosphere on the biochemical, physical and general quality characteristics of various vegetable products is in progress.

New product research with vegetables is directed toward development of "quick cooking" peas and beans; beet chips; Puerto Rican style soups; snack items; and new sauerkraut products. Methods of processing, product characteristics and storage stability are determined. Some pilot plant research is done, but basic principles relating to composition, quality and functional properties are emphasized. Product characteristics such as sweetness, concentration of individual sugars, rheological properties, softness, water absorption, color and pigment are related to organoleptic properties and consumer acceptability.

The total station scientific research effort devoted to vegetable processing and products is 46.4 professional man-years.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Identification and Characterization of Flavor and Aroma Components of Pickle Products. Basic information on the lipid and fatty acid composition of fresh cucumbers and on volatile flavor components after pure culture fermentation has been obtained in cooperation with the Michigan and North Carolina Agricultural Experiment Stations and the Pickle Packers International, Inc. Eight fatty acids (palmitic, stearic, myristoleic, linoleic,

linolenic, myristic, oleic, and palmitoleic) were found in all six sizes of Model variety pickling cucumbers analyzed (from 1/2" to 2-1/2" in diameter). The saturated acid, palmitic, as well as two unsaturated acids, linoleic and linolenic, represented almost 90% of the total acid content (=0.05% on a fresh wt. basis). The unsaturated acids have been shown by others to be responsible for bleaching and off-flavor of vegetables such as peas and lima beans. Initial gas chromatographic studies of the flavor components of pure culture pickle fermentations have revealed the presence of at least 6 carbonyl compounds in the brines. Three of these--acetaldehyde, acetone, and an unidentified carbonyl--were obtained from 3 common lactic species (L. plantarum, L. brevis, and P. cerevisiae). The remaining carbonyls--iso-butylaldehyde, 2-butanone, and iso-valeraldehyde--were found in fewer brines and generally comprised less than 10% of the total carbonyl content. Brines from 4 strains of L. brevis did not contain iso-butylaldehyde or 2-butanone. After extensive work on the development of new and complex techniques using gas chromatography, analyses were extended to 34 strains of the 3 bacteria. The results indicated that the primary flavor components resulted from the species used to accomplish the fermentation process rather than from the cucumber per se; consequently, flavor of the finished pickle product may be determined largely--if not entirely--by the bacterial species chosen. However, it will require an extensive screening of homofermentative bacteria to select those strains and species giving the most palatable flavor sensations. Also in progress are companion studies on the flavor components in vinegar prepared commercially by three pickling companies and sampled at various stages of manufacture and storage and in pickle products made with each company's vinegar. Further research will emphasize qualitative and quantitative analysis of the flavor components of pure culture and natural cucumber fermentations. (S3 5-21).

B. New and Improved Food Products

1. New and Improved Dehydrated Sweetpotato Products. Information has been obtained on the enzymatic treatment of uncured sweetpotatoes with a commercial amylase to facilitate processing and to improve the characteristics of precooked dehydrated sweetpotato flake products, and on the use of additives to stabilize the processed flakes. Continued evaluations of flakes prepared from uncured sweetpotatoes with commercial amylolytic enzymes used to bring about changes comparable to curing were encouraging. Only slight deterioration was noted after 9 months' storage of the flakes in nitrogen. Several variables were later shown to affect the susceptibility of raw roots to amylolysis: time lapse between digging and processing, time of harvesting, and climatic conditions during the harvesting season. When the process was modified with respect to water conditions and level and duration of enzyme treatment, flakes produced from uncured sweetpotatoes were comparable to those made from cured and stored roots, but after one month's storage in nitrogen the flavor of the former was unsatisfactory; however, this result may be associated with the environmental history of roots used. Selected additives (e.g., high maltose or dextrose corn sirups), antioxidants (e.g., butylated hydroxyanisole or butylated hydroxytoluene), and synergists

(e.g., citric acid or ethylenediamine tetraacetic acid), either alone or in combination, failed to further improve the stability of air-packaged flakes from cured and stored sweetpotatoes; however, flakes containing these additives retained acceptable flavor after 5 months' storage in nitrogen. Future work will be directed toward developing a practical method for the enzymatic treatment of uncured sweetpotatoes and toward stabilizing the flakes made from both cured and uncured sweetpotatoes when packaged in an atmosphere of air. (S3 5-19, S3 5-25).

2. Development of Processed Celery Products of Improved Flavor and Convenience. Dehydrated celery far superior to commercially available products has been produced through use of the "explosion puffing" technique and a more efficient, high velocity dehydrator. The procedure consists of blanching, freezing, thawing, partial dehydration to about 70% moisture, puffing at 30 pounds of pressure, and drying; in this process, the most important variables to control are moisture content before puffing and puffing pressure. The new technique has solved the problem of incomplete rehydration and fiber toughness characteristic of earlier products. Although flavor loss is still a major concern, this deficiency is being investigated, particularly with respect to use of powdered leaves and recovered essences as potential sources of flavor. A highly efficient means of recovering volatile essence from steam blanched celery has been found, but extraction of essence from the steam condensate presents a problem. At the present time adsorption onto charcoal and solvent desorption offers the most promising means for concentration of the essence. Concentrated celery juice has been produced with and without added essence. It has good potency and when mixed with other vegetable juices, its flavor is easily detectable in small concentrations. However, production of a powdered celery juice presently seems impracticable because of lack of solids in the concentrate. The research has been facilitated by the development of procedures for rapid moisture determinations during dehydration runs and for assay of total flavoring capacity of processed celery products. The dehydrated celery will be subjected to tests for storage stability and to evaluation by taste panels. Future research will be directed toward exploration of various aspects of flavor retention and flavor restoration. (S3 5-23).

3. Development of New and Improved Processed Products from Texas Vegetables. Research is continuing to improve food products processed from carrots, green beans, and tomatoes. Samples of precooked dehydrated carrot flakes packaged in an atmosphere of nitrogen and stored for 11 months at 0° and 68° F. have retained their good quality. However, a sensory evaluation of the flakes packaged in air was discontinued at the end of three months' storage because the flavor had deteriorated to the point of being repulsive. Before commercial production of carrot flakes becomes feasible, it will be necessary to find a method suitable for increasing flake density.

In research on bush green beans, a number of varieties and two strains grown at two locations in Texas are being evaluated for their overall respective processing characteristics.

Other investigations demonstrated that firming tomatoes by blanching them in a 2% solution of calcium chloride increased the percentage of whole tomatoes when they were canned. The drained weight of canned tomatoes that had been blanched in the calcium chloride solution increased about the same extent whether their skins had been pricked or not. Measurements of viscosity of canned juices of Chico, La Bonita, and Homestead tomatoes correlate directly with their respective ammonium oxalate soluble fraction, inversely with the percentage of water-soluble pectic substances, and not at all with total pectin substances or total solids. The relationships noted may be significant in explaining the high viscosity of the canned juice of the Chico in comparison with La Bonita and Homestead. Whether size and shape of cellular particles also influence viscosity remains to be determined. Investigation of the effects of pectic substances on the processing characteristics of tomato varieties is continuing. (S3 5-22).

C. New and Improved Processing Technology

1. Development of Processing Methods Applicable to Commercial Production of Dehydrated Sweetpotato Products. Precooked, dehydrated sweetpotato flakes processed at SU from east-coast-grown Nemagold and Oklamor variety sweetpotatoes were evaluated by the Commonwealth of Virginia and rated "good." During periodic taste tests at SU, the product retained its flavor as long as 9 months. The tests are continuing. Results of this work have interested an industrial group in the Virginia area, which is looking into the possibilities of flake production.

Cooperative work with Milprint, Inc. and Continental Can Co. to determine the type of flexible package required for retail marketing of sweetpotato flakes has shown that a laminated pouch construction of polyethylene, foil, polyethylene, and paper or cellophane (inner to outer layer) may be suitable. However, the desired 100% test results have not yet been attained. In cooperation with ERS, 1200 samples of flakes in each of three types of packages were evaluated in a retail market test; the order of consumer preference was glass jar, flexible pouch, and then metal can.

A new project is directed toward developing stable sweetpotato flakes from sweetpotatoes of different varieties and environmental history. Numerous variables for the processing phase of enzymatic treatment of uncured Goldrush sweetpotatoes have been investigated to obtain the best flakes from this variety. Enzyme treatment before cooking resulted in a flake product that was not as good as that obtained by treatment after cooking. On the basis of current information, curing of sweetpotatoes to achieve best flake production appears to include treatment at 85° F. and 85% relative humidity for 10-14 days and storage for at least 5 or 6 weeks at about 60° F. The effect of drying variables on drying rate, bulk density, and quality of flakes was established. The maximum drying rate was found to be attained at the minimum retention time and minimum roll spacing that would produce an apparently uniform sheet along the width of the rolls. Initial tests with other varieties indicated that the processing characteristics

of Georgia Red were comparable to those of cured Goldrush but that the taste of the former was inferior; and that enzyme-treated cured Centennials processed as well as untreated cured Goldrush and had a good bulk density and a taste rated fair. Evaluation of stored sweetpotato flakes indicated that cultural practices and environmental and curing conditions have a greater effect on flake stability than do processing conditions.

Continued research on the processing of stable sweetpotato flakes has permitted SU to provide considerable technical information to two additional companies planning commercial production of the flakes. One company has installed flaking equipment and conducted test runs for the production of flakes, whereas the other plans to install a flake plant for operation during the 1964-65 season. Work to improve processing conditions and to utilize different varieties of sweetpotatoes will continue, as will cooperation with interested industrial organizations. (S3 5-19, S3 5-25).

2. Investigations to Improve Quality and Reduce Cost of Processed Cucumber Products. Recent research on pure culture pickle fermentations has brought the process closer to a practical, commercial basis. A continuous heat-shocking process for ridding the cucumbers of vegetative, asporogenous microorganisms prior to inoculation with lactic cultures was developed and successfully used in cooperating commercial plants as a step in the overall pure culture fermentation process. Preliminary results indicated that vacuum dried lactic cultures prepared in granule form by a major pharmaceutical company were not as satisfactory as either freeze-dried or broth cultures for initiating pure culture fermentations in heat-shocked cucumbers.

Recent evaluation of pure culture dill pickles 18 months old placed them at the top of the "good" category, equivalent to the rating of samples evaluated after 3 and 7 months' storage. This is significant because long shelf life is considered a very important characteristic for pickles and many other food products. In the pure culture fermentation work, sectional differences in taste preference have become evident, southern areas preferring a product more acid than that liked in northern regions. It has also been shown that good quality, pure culture dills can be produced satisfactorily either in glass jars or in tin cans. Plans for a new project include improving the present process and scaling it up for pilot scale and commercial production. (S3 5-20).

In cooperation with plant breeders and industry, new cucumber varieties that had undergone brine curing were evaluated for color, shape, percent of cure, texture, absence of bloaters, and overall acceptability for commercial use. Fresh pack dill pickles made from several cucumber varieties were also tested. This continued assistance to cucumber breeders in the proper evaluation of their breeding material is essential to the development of better varieties for the pickling industry. (S3 5-22).

PUBLICATIONS -- USDA AND COOPERATIVE PROGRAMS

Chemical Composition and Physical Properties

Gold, Harvey J. and Wilson, Charles W. III. 1963. A colorimetric ester test as a measure of total flavor in celery. Proc. Florida State Hort. Soc. 76, pp. 240-242.

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New and Improved Food Products

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AREA NO. 11 - NAVAL STORES PROCESSING AND PRODUCTS

Problem. More uses for turpentine, rosin and pine gum need to be developed through research to provide new industrial markets for current and anticipated production of gum naval stores. These gum naval stores products face serious competition for markets from research-developed products, especially those from the chemical and petroleum industries. As an illustration, turpentine has lost substantially all of its industrial solvent market to low-cost petroleum based solvents. New fundamental information about the chemistry, composition and properties of pine gum, rosin and turpentine is needed to fully exploit their unique characteristics in the production of new and improved industrial products having utility as industrial chemicals, polymers, plastics, elastomers, resins, plasticizers, surface coatings, textile finishes, odorants, insecticides and herbicides. There is also a serious need to improve existing processes and develop new processing technology for the industry.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program at Olustee, Florida, involving organic chemists and a chemical engineer engaged in both basic and applied research to discover and develop new and improved uses for pine gum and its products. In research to develop new and improved industrial products from pine gum, rosin, turpentine, or their components, conversion of the resin acids derived from gum rosin and pine gum to new polyfunctional products by reaction with suitable chemicals is under investigation to develop intermediates for production of resins, plastics, plasticizers, and other products. Another research approach involves the condensation of the unsaturated (olefinic) materials present in pine gum with certain reactive chemicals (dienophiles) to produce industrially useful chemicals. Research is also being conducted to develop uses for photosensitized oxidized pine gum and components, primarily in the fields of plastics and rubber. Other research includes investigations to convert turpentine and rosin into polymerizable products suitable for making new polymers, plastics, and resin; to prepare chemical intermediates and modified rosin compositions by hypochlorite reaction of rosin and resin acids; to convert rosin, resin acids, and resin acid derivatives to polyfunctional compounds useful in plastics, resins, and surface coatings by formaldehyde addition and subsequent reactions; and to produce reactive chemical intermediates from turpentine and terpenes derived from it by reaction with inexpensive dienophiles. The Pulp Chemicals Association supports a Fellowship at the Naval Stores Laboratory for the purpose of conducting research to develop a suitable method for determining rosin and rosin derivatives in protective coatings, a necessity if rosin is to be allowed in certain types of these coatings from which it is now excluded. Informal cooperation is maintained with other agencies and industrial firms in connection with the naval stores research program. The U. S. Forest Service cooperates by supplying selected samples

of pine gum.

Additional research on new and improved industrial products is in progress under contract at the University of Cincinnati, Cincinnati, Ohio, on the application of the oxo and related reactions to terpenes and resin acids to produce new, useful alcohols, aldehydes, and/or acids, and the characterization of the products thus obtained; at Cornell University, Ithaca, New York, on the synthesis of terpene alcohols and glycols for use in the production of new and useful terpene derived polymers; and at the University of Florida, Gainesville, Florida, on the development of a practical process for the conversion of α-pinene to dimers in good yields, and the conversion of these dimers to useful, reactive derivatives.

Research in the field of chemical composition and physical properties is in progress under a grant of P.L. 480 funds to the Juan de la Cierva School of Technical Investigations, Barcelona, Spain, for development of new or improved methods of preparing selected terpene alcohols for use as standards, to obtain basic information on the composition and properties of products made from pine gum (project duration - 3 yrs.).

The Federal in-house scientific research effort in this area totals 15.0 professional man-years. All of this effort is on new and improved industrial products. The contract research involves an additional 2.7 man-years on new and improved industrial products. P.L. 480 research involves 1 grant for research in the field of chemical composition and physical properties.

The following line of work was terminated during the year: (1) Development of process for isolation of levopimaric acid from pine gum (under new and improved processing technology).

PROGRAM OF STATE EXPERIMENT STATIONS

State stations did not report work in this area.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Composition and Physical Properties of Pine Gum. Fundamental information on the chemical transformation of olefinic terpenes by hydroboration and subsequent reactions is being developed in contract research at Purdue Research Foundation. A systematic study of the hydroboration of terpenes was undertaken to elucidate the mechanism of the reaction. Preliminary experiments were initiated with an alicyclic terpene, 3-carene. A study on the hydroboration of limonene will be initiated to obtain basic and fundamental information on the hydroboration of a diolefinic alicyclic terpene with an endocyclic and exocyclic olefinic moiety. (S4 1-112(C)).

B. New and Improved Industrial Products

1. Development of Intermediates for the Production of Resins, Plastics, and Plasticizers from Pine Gum and its Components. The preparation of chemical intermediates from naval stores products for use in industrial applications has continued. The esters and the corresponding mono and diepoxides of the esters of α-campholenyl alcohol[1-(2-hydroxyethyl)-2,2-3-trimethyl-3-cyclopentene and α-campholenic (2,2,3-trimethyl-3-cyclopentene-acetic acid) derived from α-pinene oxide], adipic, oleic, decanoic, and acetic acids have been prepared without difficulty. α-Campholenyl alcohol is easily obtainable from α-campholenaldehyde by aluminum isopropoxide or lithium aluminum hydride reduction. Esters from oleic, adipic, stearic, campholenic acids per se should make good plasticizers for PVC. With peracids, the corresponding mono and diepoxides are easily obtainable and should make good stabilizers and plasticizers for PVC. Since the epoxides react with acids to give a mixture consisting of alcohol (or ester) and ketone it seems unlikely that diepoxides will be useful for epoxy resin by the usual procedures. The epoxides, however, should undergo neutral or basic reactions to give amines, amino alcohols, glycols, and possibly polymers. A study of the chemistry of the epoxides of α-campholenyl alcohol will be completed. Evaluation of the esters and the mono and diepoxides of the esters for use as plasticizers and stabilizers will be continued. New work on forming copolymers of vinyl pinolate and vinyl chloride and vinyl acetate and study of the preparation and reaction (stability) of 2,2-dimethyl-1,3-(2-hydroxyethyl)-cyclobutane will be started. (S5 2-38).

Additional work prior to termination of the project (S5 2-40) on production of reactive chemical intermediates from turpentine by reaction with low molecular weight reagents indicated that the mixed terpenyl chlorides formed by chlorination of pinenes with hypochlorites could be used to alkylate primary amines such as butyl amine. On the basis of results from this terminated research project, the terpene-dienophile reactions have been selected as the most promising for addition of valuable functional groups to terpenes. In initial studies of the reaction of terpenes with dienophiles the separation and proof of structure of four acrylonitrile adducts, two fumaronitrile adducts, and the tetracyanoethylene adduct of α-terpinene have been completed. The mixed acrolein adduct of α-terpinene, which can be prepared in high yield, was converted to a secondary amine by reductive ammonolysis with n-butyl amine. Successful reductive ammonolysis of α-terpinene opens the way for the production of a unique series of secondary amines for evaluation in various end uses. Separation and characterization of the α-terpinene Diels-Alder adducts will be completed as rapidly as possible and emphasis shifted to conversion of the primary products to amines and other derivatives. Methods for making similar products directly from dienophiles and pinenes or limonene will be sought. (3.1 man-years per year). (S5 2-40; S5 2-48).

2. Addition of Chemicals to Rosin Acids With Emphasis on Photochemical Methods to Produce Chemicals Useful in Manufacturing Surface Active Agents, Textiles, Paper and Plastics. The utilization of photosensitized-oxidized pine gum (POPG) as a cheap source of free radicals has been successfully demonstrated in a variety of applications and should help stimulate

industrial interest in this product. A process for the preparation of POPG has been worked out in detail. Information as to the properties of POPG has been developed and is of considerable importance in the evaluation of POPG for specific industrial applications. Evaluation of POPG-diepoxide as metal salts as stabilizers for poly-(vinyl chloride) will be carried out. Work on the photosensitized reaction between levopimaric acid and sulfur will be completed. (S5 2-47).

3. Conversion of Turpentine and Rosin Acids into New Polymers, Protective Coatings and Resins. Research on the preparation of polyfunctional compounds from rosin, resin acids and derivatives by formaldehyde addition is being completed. The adduct of levopimaric acid and formaldehyde and the diol and other derived compounds are readily obtained from pine gum. Since the turpentine does not appear to be affected and the residual rosin should be usable as formaldehyde modified rosin directly or admixed with processed rosin, the present reaction offers a series of new products available without appreciable loss or degradation of saleable byproducts already known to the users of products from pine gum. It should be possible to find uses for methylolated resin acid mixtures and rosin as intermediates for new resins, polyurethanes, etc. (S5 2-43).

The development of a two-stage process for the preparation of rosin-based polyesters permits the preparation of low acid number products having greatly improved resistance to solvent and alkali. The discovery that heat polymerized rosin can be used in the preparation of high quality polyesters offers the possibility of preparing these products from higher concentrations of rosin and lower concentration of the more expensive dienophiles. The heat polymerized rosin should have a paler color and better color stability than ordinary rosin. The determination of the stereochemistry of the Diels-Alder adducts of rosin used in this work was of assistance in developing methods for large scale separation of these products. Work on preparation of polyesters from rosin will be continued with special emphasis on use of thermally polymerized rosin as one of the dibasic acids. (S5 2-42).

Studies of the polymerization of terpene derivatives from pine gum have been made in contract research at the University of Arizona. One of the octadienes derived from pine gum copolymerizes with ethylene and propylene to give an interesting polymer. The functionality introduced via the octadiene may permit crosslinking or vulcanization of the polymer. A large chemical company is evaluating the terpolymer. The experimental work under this contract is nearing completion. Basic information has been obtained concerning the relative rates of polymerization of diverse monomers derived from agricultural products and of the general value of the homopolymers and copolymers obtained. (S4 1-89(C)).

Contract research on the application of the oxo and related reactions to terpenes and resin acids to produce alcohols, aldehyde, and acids is being carried out at the University of Cincinnati. Rates of reaction of several

terpenes in the oxo reaction have been determined. Effects of solvent and temperature have been explored. In general, α-pinene and limonene react more readily than α-terpinene and myrcene. Mixtures of products are obtained in all cases. Progress has been made on separation and characterization of the products and tentative structures have been assigned to a few individual products. α-Pinene and limonene yield similar mixtures. Hydrogenation occurs along with hydroformylation. The results to date go far toward providing a basis for selecting one terpene for more intensive study and toward defining the course of the oxo reaction with terpenes. They also demonstrate the need for special conditions such as use of terpene-cobalthydrocarbonyl complexes if a double bond is to be retained or diols prepared. Future research will be on the investigation of the reaction of $HCo(CO)_4$ with terpenes in the absence of CO and H_2 and on other approaches toward getting unsaturated monofunctional products. (S5 2-45(C)).

Synthesis of terpene alcohols and glycols for use in the production of new and useful terpene derived polymers is being carried out in contract research at Cornell University. Screening reactions of α-pinene, limonene, camphene, α-terpinene, and myrcene with formaldehyde under thermal and acid catalyzed conditions have been carried out. Under thermal conditions α-pinene gives relatively high yields but complex mixtures. Camphene yields a major product in fair yield. Limonene also shows promise. Acid catalysis favors ester formation. Some of the products have been tentatively identified and others partially characterized. (S5 2-46(C)).

Research has continued on the development of a method for the determination of rosin and rosin derivatives in protective coatings in cooperation with the Pulp Chemicals Association. The Association supports a Fellowship for the research. Studies aimed at obtaining a gas chromatographic fingerprinting of the oil portion of tall oil modified alkyds have resulted in the discovery of a liquid phase which displays a remarkably good selectivity for the resin acids. The liquid phase of choice for GLC analysis of resin acid mixtures is Versamide 900 except where analysis for levopimaric acid is required. The fractionation and subsequent analysis of tall oil fatty acids containing rosin and rosin maleic adduct must be modified to include a means for determining small amounts of rosin in the adduct-containing fraction. (S5 2-39).

C. New and Improved Processing Technology

1. Processing Investigations to Produce Naval Stores Products of Improved Quality at Lower Costs. Research on the development of a process for the isolation of levopimaric acid from pine gum is being completed. Satisfactory processes have been developed for the isolation from crude pine oleoresin of levopimaric acid in the form of 2-amino-2-methyl-1-propanol salt products whose contents of levopimaric acid salt ranged up to 85%. These resin acid mixtures are of interest to the naval stores industry since they can be obtained more easily and more economically than pure materials. (S5 2-41).

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New and Improved Processing Technology

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AREA NO. 12 - PROCESSING AND PRODUCTS - SUGARCANE

Problem. Quotas established by the Sugar Act effectively prevent the accumulation of surpluses by limiting production to estimated requirements at stable, and normally low prices for sugar. Prices received by farmers of the United States and Puerto Rico for sugarcane are based upon the recoverable sugar content of the cane; and the rising costs of production and processing make imperative the more efficient recovery of increased amounts of sugar to provide adequate returns for both processors and growers. Currently recovery of 75% of the total sugar in the cane is considered satisfactory in Louisiana, and about 83% in Puerto Rico and Hawaii. Improved processing methods could increase the recoverable sugar to at least 85% in Louisiana and over 90% in other areas. The development of more efficient processing methods depends in turn upon the acquisition of adequate data on the quantitative composition of juices extracted from sugarcane, and of materials processed to recover sugar. The chemical industry provides a promising potential for the utilization of additional sugar since more than 40 billion pounds of chemical products are produced annually and sold to every section of American industry. More information is needed on the chemistry and properties of products from sugar to expand their utilization and on the application of these derivatives in the production of plastics, protective coatings, emulsifiers, detergents and the like.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving at the Southern Utilization Research and Development Division organic chemists, physical chemists, and chemical engineers engaged in basic research on the composition and properties of sugarcane, sugarcane juices and derived products, and in applied research directed to the development of new and improved sugarcane processing technology.

Basic and exploratory studies are carried out at New Orleans, Louisiana, on the composition of sugarcane and sugarcane juices as a basis for developing more efficient methods for economical production of high grade end products. Present emphasis is on the composition and properties of the "gums" and other complex, non-sugar polysaccharide constituents which reduce the recovery and impair the quality of raw sugar. Materials used in this research are being obtained from cane processed for pilot-plant experiments in cooperation with the American Sugar Cane League. In cooperation with the Cane Sugar Refining Research Project, Inc. -- an association of commercial sugar refiners that maintains three research Fellowships at the Southern Division -- research is also in progress to investigate the fundamental chemistry and physics of processes employed in the refining of raw sugar, and of novel methods of purification devised to improve the efficiency and economy of sugar refining.

Research on new and improved processing technology is being conducted at New Orleans, Louisiana, the U. S. Sugarcane Products Laboratory, Houma, La., and the Audubon Sugar Factory (Louisiana State University), Baton Rouge, Louisiana, to develop on a pilot-plant scale novel and more effective means of clarifying sugarcane juice, and improved methods of processing and purifying sirups to obtain greater recovery of raw sugar of higher quality at lower costs. This research is planned and conducted in close cooperation with the American Sugar Cane League and individual sugar companies. Sugarcane for the work is furnished by the League and use of the Audubon Factory for milling of the cane through the cooperation of Louisiana State University. Cooperation is also maintained with the Crops Research Division, ARS (U. S. Sugarcane Field Station, Houma, La.). Informal cooperation is maintained with the industry in evaluating quality of raw sugar and economic aspects of new processing methods.

Other research on chemical composition and properties has been initiated under a grant of P.L. 480 funds to Kyoto University, Kyoto, Japan, for isolation and identification of the nucleic acid derivatives of cane molasses, in order to obtain information applicable to expanding the utilization of molasses industrially and in feeds (project duration - 2 yrs.).

The Federal in-house scientific effort at the Southern Division devoted to research in this area totals 11.3 professional man-years. Of this total 4.4 are devoted to chemical composition and physical properties and 6.9 to new and improved processing technology. P.L. 480 research involves 1 grant for research on chemical composition and physical properties.

PROGRAM OF STATE EXPERIMENT STATIONS

Basic and exploratory studies are being carried out at the Puerto Rico station to determine by the use of ion-exchange procedures the possibility of producing sugars that may be utilized in the production of hard candies and similar products without further purification. Experiments will be carried out in pilot-plant scale to evaluate scaling-up the process and to determine operating costs.

Research is also in progress to develop pilot-plant fermentation procedures for use in fermenting as efficiently as posssible molasses mashes to produce high quality rums. Other work centers around development of pilot-plant distillation procedures for use in distilling fermented mashes and development of analytical procedures for quality appraisal of rums. A continuous search for new strains of yeast suitable for the fermentation of blackstrap molasses and other materials derived from sugarcane is conducted.

In cooperation with the USDA, several storage and other experiments pertaining to the quality of sorgo juice for sirup and sugar production are conducted annually at the U. S. Sugar Plant Field Station, Meridian, Mississippi. Chemical studies center around total sugars, dextrose, levulose, sucrose and nitrogenous components.

Indiana research seeks to synthesize analogues of important metabolic sugars wherein hetero atoms such as sulfur, selenium or nitrogen replace the normal ring oxygen atom. Sugar analogues and their derivatives will be tested for usefulness as a medicine or as agricultural chemicals.

The research effort on utilization of sugarcane is 3.9 professional man-years.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Basic Studies of the Chemical Composition and Physical Properties of Sugarcane Juice and Its Products. More complete characterization of the alcohol-precipitable, water-soluble gum constituents from reconstituted raw cane juice solids was obtained by determinations of the pentoses as well as the hexoses in hydrolysates. Glucose predominates in all of the preparations analyzed; together with percentages of fructose, galactose, arabinose, xylose, and rhamnose, 82.5% of the total composition of one preparation has been accounted for. Analyses of preparations from thirteen different samples of juice solids provide evidence that some of the juices obtained in pilot plant processing experiments contain dextran, which is not separated readily from the gum. A large, uniform supply of gum as free of dextran as possible has been obtained from two tons of cane specially handled and processed promptly in a small, clean mill. This material will be used in research continuing under a recently initiated project on fundamental investigation of the soluble, non-sugar polysaccharides of sugarcane. An individual polysaccharide has been isolated from the gum complex. It has a specific rotation of -43°, a molecular weight in excess of 100,000, and constituent sugars consisting of glucose, arabinose, xylose, fructose, and rhamnose.

Its isolation is a major step toward the elucidation of the nature of the high molecular weight, alcohol-precipitable substances of sugarcane juice, which reduce the recovery and impair the quality of raw sugar. Characterization of this levorotatory polysaccharide will make it possible to develop not only a method for determining its presence in juices and syrups but also a purification process to reduce its concentration before the sugar is crystallized. (S5 1-71).

Under a P.L. 480 grant at Kyoto University, Japan, research to isolate and identify the nucleic acid derivatives that occur in sugarcane molasses is in its initial stages. Progress is being made in applying techniques such as thin layer chromatography, ion exchange chromatography, and continuous liquid-liquid extraction to the isolation of nucleic acid derivatives. There is evidence that cytosine, cytidine, uracil (or uridine), adenosine and adenine are present in cane molasses; they are presently being isolated and further identified. Later, the research will be extended to a study of the occurrence of nucleic acids in cane juice. The basic information obtained in the project is expected to be useful in assessing the role of

minor non-sugar components in sugarcane processing and in the use of cane molasses for feed and industrial applications. (UR-A11-(50)-7).

2. Investigations of the Fundamental Chemistry and Physics of Sugar Refining. In cooperation with the Cane Sugar Refining Research Project, Incorporated, an association of commercial sugar refiners, research under a new project is being directed toward improving the recovery of sugar through basic studies of the chemistry and physics of refining operations. Reduction of losses by only one percent would result in a gain of approximately 15 million dollars per annum to the domestic sugar industry, and like savings may be realized from reduction in costs through automatic and more exact control of refining. This fundamental research on the basic chemistry and physics of the refining process for sugar will foster better understanding of the clarification process. Facets of the project include measurement of the molecular weight of sugarcane gums, a new method for the specific determination of glucose, and analysis of clarifier scums. (S5 1-77).

B. New and Improved Processing Technology

1. Processing Procedures to Improve the Refining Quality of Raw Sugar. Pilot-plant development of processes to improve the refining quality of raw sugar is continuing under a new project. The USDA pilot plant for juice clarification was used to capacity during the season to determine optimum dosage rates for two flocculants in clarification and to measure clarification efficiencies on commercial canes harvested mechanically and by hand. This work demonstrated flocculant NALCO D-1782 to be equal to Separan AP-30, which has been used commercially for several years, both performing well at 2 to 6 p.p.m. on machine- and hand- harvested crops and improving clarified juice quality for cane harvested during all kinds of weather. Tests on the newest commercial variety, C.P. 55-30, provide assurance of satisfactory processing in expanded cultivation. Relationships between filterability, content of insolubles, and refinery performance of selected raw materials were determined in a refinery through the first three processing steps of affination, clarification, and filtration. Refiners and raw sugar producers have evinced great interest in the simple and rapid Millipore filterability test, which determines the efficiency of removal of insolubles at each step, indicates the raw sugar best suited to a given refinery, and may provide an additional criterion of raw sugar quality. (S5 1-80, Pending).

PUBLICATIONS -- USDA AND COOPERATIVE PROGRAMS

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AREA NO. 13 - REPLACEMENT CROPS - UTILIZATION POTENTIAL

Problem. Farmers could achieve more economic use of their land if new and profitable crops were available for their choice that would have different end-use patterns from those presently grown. For example, it would be advantageous to develop a new oilseed crop yielding unique fatty acids that could find industrial use in applications for which acids from presently available domestic oilseed crops are unsuitable. To develop a new crop, three basic steps are involved: (1) Survey of wild plants, in cooperation with plant scientists, to identify those having both potentially valuable components and promising agronomic potential for use in the U. S.; (2) detailed physical and chemical characterization of components of interest to obtain clues to likely end uses; (3) selection of the most promising species followed by additional utilization research to explore uses and demonstrate industrial potential and by additional agronomic research to establish proper cultural practices and select the best strains and varieties. Only after these steps have been successfully accomplished can a proposed new crop be offered to agriculture and industry for introduction and development. Obviously, a program of this type is a long-range one. Yet, whether the future of agriculture involves conditions of surplus, of greater emphasis on foods and feeds, or of necessity for greater national self-sufficiency, the nation will benefit from availability of optimum, practical crop plants to serve its needs.

To achieve the objective, survey and characterization work needs to be greatly increased, since the greater the number of species examined, the greater will be the opportunities for finding plants meeting the criteria of high utilization and agronomic potential. Work of the Department has already revealed several promising sources of new potentially valuable water-soluble gums, pulp fibers, and oils containing unique fatty acids such as hydroxy unsaturated acids, capric acid, epoxidized acids and unusual long-chain fatty acids. In order to demonstrate the potential of these new materials, further work is required on their physical and chemical properties and reactions, on processing to obtain maximum recovery from source plants and on byproducts from processing, such as oilseed meals.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving organic and analytical chemists engaged at New Orleans, Louisiana, in research to develop and evaluate industrial chemical products from the oils of certain new oilseed crops having production potentials as replacement crops. Oils from the seeds of the plants Limnanthes and Cuphea, rich in unusual long-chain unsaturated acids and capric acid, respectively, and from seeds of Umbelliferae such as parsley, carrots, fennel, dill, and coriander containing high percentages of petroselinic acid, are currently being investigated. The research is concerned with chemical modification of the oils and their

fatty acids to produce materials having potential utility in plastics, plasticizers, synthetic rubbers, protective coatings, and other industrial products. Research has been initiated to determine the chemical characteristics of juices obtained from selected new varieties of sweet sorghum canes grown in the Rio Grande Valley of Texas; and to determine the effects of cultural and harvesting practices on these chemical characteristics in relation to suitability for sugar recovery.

Close cooperation is maintained with the New Crops Research Branch, Crops Research Division, in the procurement of seed and in joint evaluation of the potential of the new crops. The Pharmacology Laboratory of the Western Division, Albany, California, performs tests as needed to determine the physiological properties of the oils, their derivatives, and the meals. Louisiana State University cooperates by testing some of the chemical derivatives for antimicrobial activity. Other appropriate agencies in the Department of Agriculture and the State Agricultural Experiment Stations cooperate by evaluating the utility of some of the new compounds prepared from the oils. Informal cooperation is also maintained with industrial firms for evaluations of promising materials developed in the research. In research on sorghum cooperation is maintained with substation 15, Texas Agricultural Experiment Station, Weslaco, Texas, and Crops Research Division, ARS.

The Federal scientific effort at the Southern Division devoted to research in this area totals 8.0 professional man-years. Of this total 2.3 is devoted to chemical composition and physical properties and 5.7 to industrial utilization.

PROGRAM OF STATE EXPERIMENT STATIONS

Discovery and preservation of valuable plant germ plasm is a continuing objective of the station program in new crops. Much of the research in this area is being done via four regional projects and in cooperation with regional centers. A large portion of the work is cooperative with USDA. Each year many plant introductions are grown and evaluated. Annual and perennial crops possessing potential for industrial or agricultural use are further evaluated for agronomic and chemical qualities. These include crops for paper pulp, drugs, insecticides, polysaccharide gums, and oils rich in acids of unusual structure. Assay of native and introduced tropical plants for products of economic value receives special attention.

Basic aspects of this program involve study of the biochemical and physiological bases for differences in crop plants. Attempts are made to determine if differences in biochemical or physiological processes can be associated with particular factors related to quality. Information concerning carbohydrate transformations is sought through study of carbohydrate formation and enzyme mechanisms.

Horticultural specialty crops are gaining in importance. A number of studies are underway to facilitate rapid development of this industry.

The total scientific effort devoted to replacement crops is 9.2 professional man-years.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Industrial Utilization

1. Industrial Products from Oilseeds Containing Capric Acid or Unusual Long-Chain Unsaturated Acids. Seeds of the potential replacement crops Cuphea, Limnanthes, and Umbelliferae are being studied with respect to the chemical modification of their oils and fatty acids to produce materials used in plastics, plasticizers, synthetic rubbers, protective coatings, and other industrial products.

In work on chemical derivatives of capric acid (the major constituent of Cuphea seed oil), treatment of 2-bromodecanoyl bromide with potassium hydroxide in alcoholic solutions afforded products (2-decenoic, 2-hydroxydecanoic, and 2-ethoxydecanoic acids) that may have antimicrobial activity. In preliminary tests, the former two agents have shown good inhibition against several organisms. A number of chemical derivatives of capric acid and other medium-chain acids have been prepared in sufficient quantity for evaluation as pesticides by the Entomology Research Division. Included are 4-(2-octenoyl)-, 4-(2-nonenoyl)-, 4-(2-decenoyl)-, 4-(2-dodecenoyl)-, 4-(2-bromodecanoyl)morpholides, and propargyl 2-bromodecanoate.

Research on isolating the component acids of Limnanthes douglasii oil has been facilitated by development of a method for the rapid gas-liquid chromatographic analysis of the methyl esters of the component acids. Methyl eicosenoate -- the methyl ester of the principal component acid -- has been produced in a 90% pure form from the mixed methyl esters by distillation techniques, and methyl docosadienoate has been isolated by means of fractional crystallization-adsorption chromatography. No catalysts have been found which are significantly more effective than p-toluenesulfonic acid for the lactonization of eicosenoic acid.

The preparation of nitrogen-containing derivatives of petroselinic acid, a major acid of Umbelliferea seed oil, has been improved and a continuous process devised in which petroselinic acid is subjected to ammonolysis and further modified to yield the final product, such as petroselinylamine or its derivatives, N-petroselinyl-1,3-propylenediamine, and N,N-bis(hydroxyethyl)petroselinylamine, without isolation of the intermediate. These compounds will be examined for their antimicrobial activities and also evaluated as corrosion inhibitors. A method was developed for preparation of the vinyl ester of the adduct of petroselinic acid and hexachlorocyclopentadiene. The ester is to be tested as a copolymer for vinyl chloride in plastics. Attempts to separate the aldehydes obtained from reductive ozonization of methyl petroselinate by fractional distillation gave more than one-half of the lauraldehyde practically free of

adipaldehydate, but all adipaldehydate fractions contained some lauraldehyde. The development of a good method of separation is important since many of the suggested uses for petroselinic acid depend on the reactions of these aldehydes. A method for determining percent petroselinate in the presence of oleate, based on reductive ozonization of the methyl esters and analysis of the resulting aldehydic fragments by gas liquid chromatography, appears promising for application to the analysis of fennel seed acids. A potentially useful polyol, trimethylol undecane, has been prepared in 30 to 35% yield by reductive ozonization of petroselinic acid and subsequent treatment with alkali and formaldehyde. Although the present project has been terminated, promising leads will be investigated under a new project. (S5 5-45).

PUBLICATIONS -- USDA AND COOPERATIVE PROGRAMS

None

AREA NO. 14 - RICE PROCESSING AND PRODUCTS

Problem. The productive capacity of U. S. rice growers has increased faster than domestic and export consumption over the past decade, thus limiting the income potentially available from this major world food grain. Detailed knowledge of chemical composition and physical properties, as related to processing is needed to guide milling, processing and product development of U. S. rices so that they can better meet the quality and new product requirements needed for expanded markets. New and diverse food products from rice that are easy to prepare, have flavor and texture appeal, and are economical to manufacture, are needed to increase the total consumption of rice both domestically and abroad. Additional needs include the development of improved milling machinery and techniques, primarily to increase the yield of head rice; intensified research on deep milling to evaluate and utilize the products, protein flour and residual kernels; and research to provide greater flexibility in the industry by developing from either medium or long grain rice new products that will provide on cooking either discrete kernels or a gelatinous food.

USDA AND COOPERATIVE PROGRAM

The Department has a continuing long-term program involving at New Orleans, Louisiana, biochemists and analytical chemists engaged in basic and exploratory studies on the distribution of the chemical constituents of milled rice in consecutive layers of the kernel with special emphasis on nutritionally important constituents such as proteins, amino acids, starch, lipids, vitamins and minerals; and on the cooking and chemical characteristics of the kernels remaining after differential removal of these layers. Findings from this research will provide the necessary basis for evaluating the economic feasibility of using high-protein rice flours (layers removed by deep milling) for protein fortification of foods and as dietetic or other specialty type foods.

Close cooperation is maintained, under formal memoranda of understanding, with the Louisiana, Arkansas and Texas Rice Experiment Stations, who supply rice samples of known variety and cultural history for the experimental studies. The Rice Inspection Service, Grain Division, AMS, New Orleans, Louisiana, cooperates by providing assistance in grading rice samples from the research investigations. Cooperation has been initiated with the Western Division.

The Federal scientific effort at the Southern Division devoted to research in this area totals 3.6 professional man-years. The present effort is on chemical composition and physical properties.

The following line of work was terminated during the year: (1) Investigation of the biochemical characteristics of rice as affected by and in

relation to age and processing characteristics, with special emphasis on the susceptibility of rice starch to amylolytic action (under chemical composition and physical properties).

PROGRAM OF STATE EXPERIMENT STATIONS

The program on rice involves evaluation of new rice varieties and lines for specific uses through cooperation with the Regional Rice Quality Laboratory. Early emphasis is placed upon developing and applying rapid and simple testing procedures for screening the selections. Subsequently, the influence of cultural methods, drying procedures and storage upon processing and product quality is determined. Basic compositional and other data relative to the quantity and quality of the proteins, lipid and starch as well as methods of parboiling are obtained.

The effectiveness of infrared drying of rough rice, as measured by rapidity of drying and maintenance of melting quality, is being studied. Results to date indicate that infrared-dried rice may deteriorate less rapidly in storage. This study will be expanded to include other varieties and the effect of maturity at harvest.

Fundamental studies on the chemistry of rice are directed toward study of the variation in the constituents of several varieties. Both chemical and physical properties are observed and related to differences in quality of rice.

New product research centers around development of effective ways to use rice in quantity food service. Products are developed and evaluated for acceptability, ease of preparation and costs.

Extended utilization of rice will be facilitated by better characterization and utilization of its proteins. This is especially true in the underdeveloped countries where rice is the principal food. Continued study of the supplementary value of high protein foods for rice and its by-products and of rice proteins for those of corn and wheat is in progress. The amino acid composition of rice is being determined since it may vary with variety and other environmental factors. The biological value of the proteins of rice when used with multipurpose food is also being investigated.

The total State scientific effort devoted to utilization of rice is 1.9 professional man-years.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Studies of Chemical and Physical Changes in Rice as Affected by Aging and Processing. Basic studies of the changes accompanying the aging of rice have been completed for a second year's (1962) crop of Bluebonnet-50 and Nato

rices. Both varieties were stored at ambient temperature (77° F.) and at 40° F. In general, the results confirm findings from the previous year's crop. A definite pattern (progressive decrease) of native alpha- and beta-amylase activities was found for the rice aged over the 10-month period. Susceptibility of the rice starch to introduced alpha- and beta-amylases reached a maximum and then decreased. As a result of this study, it has been observed that the aging process can be arrested by storage at sub-zero temperatures. In addition, indications are that by inactivating enzymes in the rice by heat or enzyme poisons, changes in pasting characteristics and culinary quality can be effected in the rice similar to those associated with aging. This fundamental information was utilized in the development of a laboratory-scale method of heat-treating freshly harvested rice to effect an improvement in the culinary properties of the rice similar to that obtained by conventional aging of the rice for 10 months or longer. Preliminary investigation of the heterogeneous distribution of protein in the rice endosperm appears to be so promising that the work on aging has been discontinued and the effort transferred to the newer approach. (S1 4-12).

2. Investigation of the Distribution of Chemical Constituents in the Rice Kernel. Extending the observations of Spanish workers engaged in P. L. 480 research, Southern Division scientists have determined that the gradient of protein concentration in brown and commercially milled rices increases from the center to the periphery of the kernel. For Bluebonnet-50 brown rice (protein content = 8.8%), removal of 7 consecutive layers, totalling 38% of the kernel by weight, indicated that the protein content of each layer was greater than the average protein content of the original kernel. Protein content of fractions 1 through 7 ranged from 21% for fraction 2 to about 10% for fraction 7. The general distribution was similar for Nato and Caloro brown rices, as well as for the milled rice sample. High protein-bearing layers were also demonstrated in wheat, sorghum grain, brown glutinous rice, and "brokens" from commercially milled rice.

To obtain the requisite removal and isolation of successive fractions of the rice as a fine flour rich in proteins, ingenuous laboratory equipment and techniques were developed, employing the principle of tangential abrasion. The equipment causes only minimal breakage of residual kernels, which are equivalent to commercially milled rice in cooking tests and have a more attractive appearance. A prototype mill that can be used for the differential milling of larger quantities of rice and other grains has been constructed, and a preliminary evaluation of its operating variables has been made. This deep milling process appears to offer considerable potential for the production of a high protein rice flour that may be a valuable food fortifier or supplement.

Future plans include investigation of the content of amino acids, vitamins, and other nutritionally important constituents, in consecutive layers and resultant residual kernels of differentially milled rices of several varieties and grain types. (S1 4-13, Pending).

PUBLICATIONS -- USDA AND COOPERATIVE PROGRAMS

Chemical Composition and Physical Properties

Hogan, Joseph T. 1963. Rice research at Southern Laboratory. Rice J. Ann. 66(7), pp. 38-41.

Hogan, J. T., Normand, F. L., and Deobald, H. J. 1964. Method for removal of successive surface layers from brown and milled rice. Rice J. 67(4), pp. 27-34.

Roseman, A. S. and Hogan, J. T. (SURDD); Stone, R. B. and Webb, J. C. (Agr. Eng. Res. Div., USDA, Univ. Tenn.). 1963. Gas plasma irradiation of rice. III. Influence on brown rice and rice bran. Cereal Chem. 40, pp. 568-575.

AREA NO. 15 - PEACH PROCESSING AND PRODUCTS

Problem. The peach industry in the Southeastern United States is dependent to a large extent on the fresh market. For example, in the South Atlantic States in 1962, 15,195,000 bushels of peaches were produced of which 12,237,000 bushels were sold on the fresh market; slightly less than 2,000,000 bushels were processed. A peach processing industry is needed in the Southeastern States to provide a profitable market for more of the edible peaches which do not meet fresh market standards and to rapidly convert a higher proportion of the overall crop to stable forms. Basic information, not now available, on the flavor components of peaches is needed to guide development of improved processed products from southern grown fruit.

Climatic conditions which favor rapid deterioration of fresh peaches both on and off the tree, erratic ripening periods and markets, and short lived peach orchards, are other factors contributing to the need for more extensively integrated fresh market-processing operations. There are technical problems preventing the more rapid development of the peach processing industry in the Southeastern States which must be overcome. Many of the peach varieties grown in the southeast require a modification of processing procedures to make satisfactory standard-type products. Still other varieties will not make standard-type products and new food forms must be found for them. Recent rapid advances in food science and processing technology make it possible through research to develop both new and improved peach products. These are needed to bolster the economics of the South's peach industry, as well as to provide the superior qualities, and greater convenience in food products, which the consumer now demands.

USDA AND COOPERATIVE PROGRAM

The Department has a program of basic and applied research on peaches being conducted under contract at the Georgia Agricultural Experiment Station, University of Georgia, Experiment, Georgia. Food chemists and food technologists conduct this research. Research to develop basic information on chemical composition and physical properties of peaches, particularly varieties grown in the Southeastern States, is in progress under one contract. Specifically, the objective of this research is to isolate, identify, and characterize the constituents of peach flavor and aroma, and acquire information needed to guide development of improved processed products from the fruit. Another contract, in the field of new and improved food products and processing technology, is concerned with research to develop optimum procedures for the production and preservation of puree and clear juice peach concentrates; to develop optimum procedures for the preparation and the handling under simulated commercial conditions of refrigerated fresh peach slices; to develop optimum procedures for canning Southeastern peaches; and to conduct experiments directed to the development of partially

dehydrated pasteurized peach products. Evaludation of different varieties of peaches, and of different processing variables are phases of the investigations. This research is carried out with the support of the Area Redevelopment Authority of the Department of Commerce.

The contract research involves a total level of effort of 2.0 man-years, 0.8 being on chemical composition and physical properties and 1.2 on new and improved food products and processing technology.

PROGRAM OF STATE EXPERIMENT STATIONS

No report for this area.

PROGRESS -- USDA AND COOPERATIVE PROGRAMS

A. New and Improved Food Products and Processing Technology

1. Development of New and Improved Processed Products from Southeastern Peaches. New and improved processed products made from the 1963 crop of southeastern peaches by the contractor (Georgia Agricultural Experiment Station) have now been evaluated. For canned peaches, numerous variables have been investigated: for example, variety, postharvest treatment, conditions of processing, and degree of ripeness. Evaluation of the effect of these variables established criteria for canning to produce good quality products. It is anticipated that chilled peach sections and a 2-fold puree-type concentrate will be prepared on a commercial scale and market tested in 1964. Fully ripe peaches of both freestone and clingstone varieties can be used for the concentrate. A clear juice concentrate is suitable for disposition of cull peaches and varieties that cannot be utilized for other products. In the 1963 season, approximately 15,000 gallons of 60° Brix concentrate were produced commercially, primarily for use in wine. It is being tested further for other applications, for example, as a topping for ice cream and desserts and as a flavoring for waffle syrup. Research to develop a partially dehydrated pasteurized peach product has not yet been as successful as for the other products. The present research has demonstrated that high quality products can be prepared from southeastern peaches; the major research effort in the 1964 season will be on canned peaches and chilled sections, with continuing work on the pureed, dehydrated, and concentrated products. (SU-0-0-1 (DC)).

PUBLICATIONS -- USDA AND COOPERATIVE PROGRAMS

None

Line Project Check List - Reporting Year July 1, 1963 to June 30, 1964

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
S1 4-	Rice Utilization Investigations-Southern Region			
S1 4-12	Investigation of the biochemical characteristics of rice as affected by and in relation to age and processing characteristics, with special emphasis on the susceptibility of rice starch to amylolytic action.**	New Orleans, La.	Yes	14-A-1
S2 1-	Cotton Utilization Investigations			
S2 1-154	Development of a Bale-Breaker-Blender for opening and blending cotton.**	New Orleans, La.	Yes	3-A-1
S2 1-156	Development of weather- and rot-resistant cotton fabrics.**	New Orleans, La.	Yes	5-A-1
S2 1-157 (C)	Design and development of acceptable cotton crepe apparel fabrics to compete with synthetic fibers in these markets.**	Philadelphia, Pa.	Yes	5-F-1
S2 1-163	Development of optimal structures for cotton fabrics for wash-wear products.**	New Orleans, La.	Yes	4-B-2
S2 1-164 (Rev.)	Development of a prototype machine for removing short fibers from cotton.	New Orleans, La.	Yes	3-A-3
S2 1-168	Exploration of cellulosic crosslinks capable of being broken and reformed at will.**	New Orleans, La.	Yes	1-B-1
S2 1-170 (C)	Investigation of the relationships between ease-of-care performance and the geometry of cotton fabrics.	Dedham, Mass.	Yes	4-B-2
S2 1-173 (C)	Determination of the mechanics of nep formation in cotton during textile mechanical processing.**	Cambridge, Mass.	Yes	1-B-4
S2 1-174	Microscopical investigation of reaction products in chemical modifications of cotton fibers.**	New Orleans, La.	Yes	1-A-2
S2 1-175 (C)	Fundamental investigation of the effects of specific type finishes on soiling of and soil removal from cotton.**	Washington, D.C.	Yes	5-B-1
S2 1-176	Preparation of cotton products containing radiation-induced polymers having desirable physical properties.**	New Orleans, La.	Yes	1-B-2
S2 1-178 (C)	Large-scale spinning evaluation of the effect of fiber properties and spinning variables on yarn properties and end breakage during spinning.	Auburn, Ala.	Yes	2-A-1
S2 1-179	Development of optimum processing procedures to minimize the detrimental effects of short fibers in cotton spinning performances and product quality.**	New Orleans, La.	Yes	2-A-2
S2 1-180	Modification of cotton with fluorochemicals to impart durable water and oil repellency.**	New Orleans, La.	Yes	5-B-1
S2 1-181 (Rev.)	Improvement in the bulk resilience and cohesion of cotton batts as a means of enhancing cotton's competitive position in this market.	New Orleans, La.	Yes	5-E-3
S2 1-182	Evaluation of the swelling behavior of cotton fibers in various environments by observation of the untwisting of single yarns.**	New Orleans, La.	Yes	1-A-1
S2 1-183 (C)	Investigation of the effects of mechanical treatments prior to, during, and following resin finishing on the ease-of-care properties of fabrics and garments.	Raleigh, N.C.	Yes	4-B-1
S2 1-184	An engineering study of the feasibility and practicality of chemical and/or resin treatment of roving by continuous processing as an intermediate step in the mechanical processing of cotton.**	New Orleans, La.	Yes	5-F-1
S2 1-185	Basic investigations to characterize fiber damage in mechanical processing from opening through carding to provide information needed to develop improved textile machinery and processing methods.**	New Orleans, La.	Yes	2-A-2

** Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Area & Subheading
S2 1-186	Chemical attachment of reactive compounds to cotton cellulose by means of polyfunctional reagents.**	New Orleans, La.	Yes	1-B-1
S2 1-188	A fundamental investigation of the drying of chemically modified cotton, with emphasis on resin treated cotton, as a means of producing cotton products of superior quality.**	New Orleans, La.	Yes	1-B-4
S2 1-189	Investigation of wet and dry crease recovery mechanisms in wash-wear cotton products.**	New Orleans, La.	Yes	4-A-1
S2 1-190	Exploratory investigations to impart improved properties to cotton needed for specific end uses.**	New Orleans, La.	Yes	5-C-1
S2 1-191	Investigation of various finishes with respect to soiling and soil removal from cotton.**	New Orleans, La.	Yes	5-B-1
S2 1-193 (Rev.)	Development of stretchable-type cotton yarns and fabrics that will successfully compete with fabrics made from synthetic fibers and blends of synthetic fibers for clothing, household, and industrial uses.	New Orleans, La.	Yes	5-E-1
S2 1-194	Exploratory investigation of methods for imparting durable luster and related appearance characteristics to cotton textiles.**	New Orleans, La.	Yes	5-D-1
S2 1-195	Investigation of radiochemical yields of high-energy radiation activated reactions of cotton to develop improved cotton products.	New Orleans, La.	Yes	1-B-2
S2 1-196	Fundamental study of mechanisms of cellulose etherifications.	New Orleans, La.	Yes	1-B-3
S2 1-197 (C)(Rev.)	Evaluation of stretch type cotton yarns in knit wear.	Raleigh, N.C.	Yes	5-E-1
S2 1-198	Relationship of fiber properties to fabric behavior in wash-wear treatments.	New Orleans, La.	Yes	4-A-1
S2 1-199 (C)	Exploratory investigation of the reaction of acetylene and related compounds with cotton cellulose.	New York, N.Y.	Yes	1-B-1
S2 1-200 (C)	Development of weather-resistant, water-repellent finishes for cotton.	Denton, Texas	Yes	5-A-1
S2 1-201	An investigation of the interfiber frictional force and associated fiber properties to improve the processing of cotton products.	New Orleans, La.	Yes	1-A-5
S2 1-202	Exploratory investigation to impart special properties to cotton through the use of lead compounds.**	New Orleans, La.	Yes	1-B-1
S2 1-203	Investigation of the effects of time and environmental conditions on the rate of wrinkle recovery of wash-wear cotton fabrics.	New Orleans, La.	Yes	4-A-1
S2 1-204 (C)	The aerodynamic separation of lint cotton into individualized fibers to provide information needed for improving cotton textile processing equipment.	Westbury, L.I., N.Y.	Yes	3-A-4
S2 1-205 (C)	The development of cotton knit fabric having increased bulk, warmth, and dimensional stability.	Clemson, S.C.	Yes	5-E-1
S2 1-206 (C)	A determination of the structural components of the cotton fiber that contribute most to tensile strength and how they can be utilized to increase tensile and recovery properties to produce cotton products having enhanced physical properties.	South Pasadena, California	Yes	1-A-2
S2 1-207	Determination of the relative effect of fiber properties on both yarn properties and spinning performance to utilize cotton more efficiently.	New Orleans, La.	Yes	2-A-1
S2 1-208	Investigation of the effects of gross and fine structures of the cotton fibers on their physical behaviors.	New Orleans, La.	Yes	1-A-2
S2 1-209	Microscopical investigations of absorption phenomena in native, mercerized, and modified cottons.	New Orleans, La.	Yes	1-A-1

** Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Area & Subheading
S2 1-210	The crosslinking of various physically modified crystalline forms of cotton as a means of producing resilient cotton textiles having improved appearance and durability to wearing.	New Orleans, La.	Yes	1-B-1
S2 1-211	Investigation of finishing treatments to produce wash-wear cotton stretch fabrics with improved strength, drape, and hand.	New Orleans, La.	Yes	4-C-1
S2 1-212	Development of test methods for stretch cotton textiles for use as a guide in producing better cotton stretch yarns and fabrics.	New Orleans, La.	Yes	2-B-1
S2 1-213	The development by chemical and mechanical treatment of stretchable cotton yarns suitable for weaving and knitting into fabrics with enhanced qualities.	New Orleans, La.	Yes	5-E-1,2
S2 1-214	Separation and identification of the cleavage products of partially etherified cottons, including crosslinked cottons.	New Orleans, La.	Yes	1-A-4
S2 1-215	Development of an improved method of feeding the cotton card to produce higher quality textile products and thus increase the utilization of cotton.	New Orleans, La.	Yes	3-A-2
S2 1-216	A study of reactions between epoxy compounds or their halohydrin precursors and cotton cellulose.	New Orleans, La.	Yes	1-B-1
S2 1-217 (C)	Effect of variation in structure on cotton fiber properties caused by environmental and genetic factors to obtain basic information important in optimum utilization of cotton.	College Station, Texas	Yes	1-A-2
S2 1-218	Development of improved instrumental techniques for selected elemental analysis of additively and chemically modified cottons to aid in improvement of cotton textile products.	New Orleans, La.	Yes	1-A-4
S2 1-219	Improved methods of etherifying cotton cellulose.	New Orleans, La.	Yes	1-B-1
S2 1-220	Investigation of improved infrared spectral techniques for the study of modified cottons to aid in the development of textile products for specific end uses.	New Orleans, La.	Yes	1-A-4
S2 1-221 (C)	Investigations to determine the effects of fiber extensibility on fiber breakage in mechanical processing.*	Knoxville, Tenn.	No	
S2 1-222 (C)	Treatment of cotton fibers by sonic energy to obtain basic information required for the development of improved equipment for processing cotton into textiles.*	Carteret, N.J.	Yes	1-B-4
S2 1-223 (C)	Effect of the soiling environment on the soiling tendency of a series of cotton finishes.*	Washington, D.C.	Yes	5-B-1
S2 1-224 (C)	Determination of optimum yarn constructions, knitting structures, and prefabrication design for producing stretchable articles of knitted cotton wearing apparel by slack mercerization.*	Raleigh, N.C.	No	
S2 1-225 (C)	The relationship of molecular size, nature, shape, conformation, and configuration of organic non-aqueous compounds to their swelling power on cotton cellulose.*	Brooklyn, N.Y.	No	
S2 1-226	The development of cotton fabrics having improved warp and filling stretch properties by a comprehensive investigation of fabric and yarn structures and processing conditions during slack mercerization and resin treatment.*	New Orleans, La.	Yes	5-F-1
S2 1-227	Excellent smooth drying and wrinkle resistant fabrics by crosslinking cotton with highly reactive methylol-amide amino acid derivatives.*	New Orleans, La.	Yes	4-B-1
S2 1-228 (C)	Investigation of the physics of seam pucker in relation to fabric structure to develop improved sewing thread for wash-wear cotton products.*	Atlanta, Ga.	No	

* Initiated during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Area & Subheading
S2 1-229 (C)	Development of a method for counting neps in cotton at various stages of textile processing.*	South Pasadena, California	No	
S2 1-230	Development of a durable wash-wear finish for cotton based upon monoalkyl carbamates.*	New Orleans, La.	Yes	2-A-2 4-B-1
S2 1-231 (C)	An investigation of the chemical modification of cotton through treatments with reagents in the vapor phase.*	East Greenwich, Rhode Island	No	
S2 1-232	The preparation of new finishing agents for cotton and cotton derivatives based upon lead and other metal compounds.*	New Orleans, La.	Yes	1-B-1
S2 1-233	Preparation of fatty acid or hindered acid esters of cotton to form new or improved end-use textile products and investigation of reaction mechanisms involved.*	New Orleans, La.	Yes	1-B-3
S2 1-234	Investigation of blending methodology to establish optimum blending procedures for maximum utilization of cottons differing widely in fiber properties.*	New Orleans, La.	No	
S2 1-235	Improvement of smooth drying properties, wet crease recovery, and moisture absorptivity in wash-wear cotton through swelling treatments.*	New Orleans, La.	Yes	4-B-1
S2 1-237 (Gr)	Investigation of fiber and yarn geometry in areas of deformation in cotton fabrics.*	Cambridge, Mass.	No	
S2 1-238 (Gr)	Correlation of surface microtopography of treated and untreated cotton fibers with resistance to soiling of cotton textiles.*	Tucson, Arizona	No	
S2 1-240 (Gr)	An exploratory study of the crosslinking of chemically modified cotton to obtain cotton fabrics with an optimum combination of resiliency and thermoplasticity.*	Princeton, N.J.	No	
S2 1-241 (C)	Investigation of factors influencing comfort in cotton apparel fabrics.*	Washington, D.C.	No	
S2 1-245 (C)	The development of weather resistant cotton textiles with improved physical properties by interfacial and graft polymerization.*	Birmingham, Ala.	No	
S2 1-247	An investigation of the chemical modification of cotton fabrics using reagents in the form of fogs or aerosols.*	New Orleans, La.	No	
S2 1-250	Treatment of cotton with fluorochemicals to produce a finish with low surface energy.*	New Orleans, La.	Yes	5-B-1
S2 1-251	Exploratory research to impart multifunctional properties to cotton through the use of specially tailored compounds.*	New Orleans, La.	No	
S2 1-252	Development and evaluation of a new machine for opening and blending bales of cotton in any desired proportion in textile mills.*	New Orleans, La.	Yes	3-A-1
S2 1-253	Wash-wear cottons of high abrasion resistance by the application of durable polymeric coatings.*	New Orleans, La.	No	
S2 1-254	Development of optimal cotton fabric structures for men's trousers and dress suits.*	New Orleans, La.	No	
S2 1-255	Investigation of effective crosslinks in cotton modified by chemical treatment.*	New Orleans, La.	No	
S2 1-256	Development of multipurpose finishes for outdoor cotton fabrics with improved physical properties.*	New Orleans, La.	No	
S2 1-258	Exploratory investigation of reversible chemical reactions to obtain information basic to the development of a commercially feasible reversible crosslink.*	New Orleans, La.	No	

* Initiated during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Area & Subheading
S3 2-	Citrus and Other Fruit Utilization Investigations - Southern Region			
S3 2-32	Investigations on the "foam-mat" drying of concentrated citrus juices to provide new citrus products of optimum flavor and high stability.**	Winter Haven, Florida	Yes	9-C-1
S3 2-34 (Rev.)	Investigation of the biochemical mechanism of the conversion of precursors to carotenoids in grapefruit as a basis for improvement of processing characteristics of and products from colored grapefruit.	Weslaco, Texas	Yes	9-A-4
S3 2-36	Investigations on composition of essential citrus oil as related to flavor of juices, concentrates, powdered juice and other products with special emphasis on essential orange oil.**	Winter Haven, Florida	Yes	9-A-1
S3 2-37	Investigations of the neutral fraction of orange peel extract for the isolation of bitter principles.**	Winter Haven, Florida	Yes	9-A-2
S3 2-38 (Rev.)	Investigation of the chemical and physical nature of components of cloud of orange juice, to provide better understanding and control of factors affecting stability of orange juice products.	Winter Haven, Florida	Yes	9-A-3
S3 2-39 (C)(Rev.)	Investigation of the effect of maturity of grapefruit on total flavonoids, naringin, and poncirin; and on the chemistry and nature of naringin and naringin-derived compounds; to provide a scientific basis for the control of bitterness in processed grapefruit products.	Norman, Okla.	Yes	9-A-2
S3 2-40	Utilization of natural and debittered grapefruit juice and puree as bases for the development of improved fruit juice blends, drinks, and concentrates.	Weslaco, Texas	Yes	9-B-1
S3 2-41	Investigations on the "foam-mat" drying of concentrated grapefruit juices to provide a new grapefruit product of optimum flavor and high stability.	Winter Haven, Florida	Yes	9-C-1
S3 2-42	Investigations of the identities, quantities and chemistry of components in Florida grapefruit responsible for excessive bitterness and harshness in processed products.	Winter Haven, Florida	Yes	9-A-2
S3 2-43	Investigations on conditions for drying as related to the storage stability and quality of "foam-mat" dried citrus products.*	Winter Haven, Florida	Yes	9-C-1
S3 2-44 (C)	Composition of flavor components of peaches, (with emphasis on existing commercial varieties in the Southeastern United States).*	Experiment, Ga.	No	
S3 2-46 (C)	Development of practical and efficient pilot plant process for the manufacture of enzymatically debittered grapefruit juice and products with improved flavor, product stability, and storage characteristics.*	Lake Alfred, Florida	No	
S3 5-	Sweetpotatoes, Cucumbers, and Other Vegetable Utilization Investigations - Southern Region			
S3 5-19	Development of a practical pilot plant process for precooked dehydrated sweetpotato flake products, with improved product quality and processing efficiency to extend the utilization of sweetpotatoes.**	New Orleans, La.	Yes	10-B-1 10-C-1
S3 5-20	Investigation of methods for the controlled fermentation of cucumbers with emphasis on the application of pure culture techniques to reduce processing costs and improve product characteristics.**	Raleigh, N.C.	Yes	10-C-2

*Initiated during reporting year.
**Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Area & Subheading
S3 5-21	Investigation of the flavor and aroma components in natural and pure culture fermented cucumber pickle products.	Raleigh, N.C.	Yes	10-A-1
S3 5-22	Investigation to develop new and improved processed products from southern grown vegetables other than sweetpotatoes and celery, including cooperative studies with federal, state, and industry agencies.	Weslaco, Texas and Raleigh, N.C.	Yes	10-B-3 10-C-2
S3 5-23	Application of new basic information on the chemical constituents of celery stalk (petiole) to the development of processed products of improved flavor and convenience.	Winter Haven, Florida	Yes	10-B-2
S3 5-25	Development of processing innovations for manufacture of stable, precooked, dehydrated sweetpotato flakes from roots of different varieties and environmental history.*	New Orleans, La.	Yes	10-B-1 10-C-1
S3 5-27	Adaptation of laboratory pure culture fermentation procedures to a commercially feasible process for the manufacture of pickled vegetable products.*	Raleigh, N.C.	No	
S4 1-	Cottonseed, Peanut and Other Oilseed Investigations - Southern Region			
S4 1-88	Investigations of solubilities of long-chain fatty acids and their derivatives important to research and industrial utilization of fatty acids from vegetable oils of Southern Region.**	New Orleans, La.	Yes	6-A-3
S4 1-89 (C)	Polymerization of vegetable oil, pine gum, and sugarcane derivatives and evaluation of properties of the polymers for use as elastomers, plastics, thickening agents, and protective coatings.	Tucson, Arizona	Yes	11-B-3
S4 1-90	New polyester products from cottonseed oil for use as edible and inedible coating materials, waxes, resins, plasticizers, and lubricants.**	New Orleans, La.	Yes	6-B-1
S4 1-92	Pilot plant scale development of a process for improving cottonseed oil color based on highly active adsorbents.**	New Orleans, La.	Yes	6-B-2
S4 1-93	Chemical modification of tung oil and its fatty acids to produce materials having utility as protective coatings, agricultural chemicals, surfactants, or plasticizers.**	New Orleans, La.	Yes	8-B-2
S4 1-99	Investigation of long-chain fatty amides and derivatives potentially useful as plasticizers, polyurethane foams, and other industrial uses.**	New Orleans, La.	Yes	6-D-1
S4 1-100	Investigations of the constituents and their modifications by processing that influence nutritive properties and consumer acceptance of processed peanut products.**	New Orleans, La.	Yes	7-A-2
S4 1-101	Engineering studies to develop a commercial process for preparing cocoa butter-like fat from cottonseed oils.**	New Orleans, La.	Yes	6-B-2
S4 1-102 (Rev.)	Development of hydrogenation techniques for cottonseed oil which will reduce cyclopropenoids with the least possible isomerization of the other unsaturated fatty acid groups.	New Orleans, La.	No	
S4 1-103 (C)	Investigations of gossypol esters and of mild oxidation products of gossypol and gossypol derivatives to develop information needed to aid the production of cottonseed meals and oils of the highest quality.	Knoxville, Tenn.	Yes	6-A-2

*Initiated during reporting year.
**Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
S4 1-104 (C)	Investigation of the chemical and physical properties of cyclopropene fatty acids in cottonseed.	Urbana, Ill.	Yes	6-A-3
S4 1-105	Investigations on the cyclopropene acid constituents in cottonseed and cottonseed products.	New Orleans, La.	Yes	6-A-2,3
S4 1-106 (C)	Investigation of the flavor and aroma components in processed peanut products.	New York, N.Y.	Yes	7-A-2
S4 1-107 (C)	Chemical investigations of cyclopropenoids to develop means of eliminating or physiologically inactivating the cyclopropenoids found in cottonseed products.	Urbana, Ill.	Yes	6-B-2
S4 1-109	Investigation of the proteins and nonglyceride lipid-soluble constituents of peanuts and processed peanut products to expand their utilization.	New Orleans, La.	Yes	7-A-2
S4 1-110	Investigations to isolate and identify the factors in cottonseed meal that cause mortalities among swine to develop information for producing cottonseed meals that can be used without restriction in feeding to any nonruminants.	New Orleans, La.	Yes	6-C-1
S4 1-111	Pilot-plant development of a cottonseed extraction process using hexane-acetone-water solvent mixtures to a stage suitable for commercial evaluation.**	New Orleans, La.	Yes	6-C-2
S4 1-112 (C)	Investigations of chemical transformations of fat and terpene olefinic compounds by hydroboration and suitable subsequent reactions to produce useful products.	Lafayette, Ind.	Yes	6-A-3 11-A-1
S4 1-113	The development of exterior and interior, intumescing, fire-retardant coatings from tung oil and tung oil derivatives.*	New Orleans, La.	Yes	8-B-1
S4 1-114	Development of methods for upgrading the quality of cottonseed oils by improving the color and eliminating undesirable components such as cyclopropene acids.*	New Orleans, La.	Yes	6-B-2
S4 1-115 (C)	Ethylene copolymerization with unsaturated fatty acid and gum naval stores compounds to extend the industrial utilization of agricultural products in commercial plastics.*	New York, N.Y.	No	
S4 1-116	Isolation, identification, evaluation, and control of fungi and toxic fungal metabolites which may develop during processing of cottonseed and peanuts to improve the acceptance of their processed products.*	New Orleans, La.	Yes	6-A-4 7-A-3
S4 1-117 (C)	Development of practical processing methods for inactivation of cyclopropene groups in cottonseed meal that decrease its value as a feed for laying hens.*	Chicago, Ill.	No	
S4 1-119 (C)	A study of the relation of the carbohydrate, amino acid and protein components of the peanut to the formation of flavor and aroma during roasting, with the objective of expanding the direct utilization of this commodity.*	Stillwater, Oklahoma	No	
S4 1-120 (C)	Development of processing methods using peanuts of known history with respect to different growing, harvesting, and curing conditions that will provide processed peanut products of high quality and free of mycotoxins.*	College Station, Texas	No	
S4 1-121 (C)	Study of the limiting environmental conditions for the elaboration of mycotoxins in peanuts, to develop information needed to assure the processing of highest quality peanuts.*	Auburn, Ala.	No	
S4 1-123	Study of rates of extraction of cottonseed with acetone-hexane-water solvent mixtures and nature and quantities of constituents in miscellas and rheological properties of marcs, to develop information basic to the production of cottonseed meal and oil of the highest quality.*	New Orleans, La.	Yes	6-C-2

*Initiated during reporting year.
**Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
S4 1-124	Preparation and evaluation of N-disubstituted fatty amides considered potentially useful as plasticizers, nitrile rubber softeners, and antifungal agents, to develop information basic to the increased utilization of cottonseed and other seed oils assigned to SU.*	New Orleans, La.	Yes	6-D-1
S5 1-	Sugars and Sirups Investigations - Southern Region			
S5 1-71	Investigation of the composition of sugarcane in relation to processing efficiency.**	New Orleans, La. and Houma, La.	Yes	12-A-1
S5 1-77	Chemical and physical investigations of sugar refining operations to improve processing of cane sugar.*	New Orleans, La.	Yes	12-A-2
S5 2-	Naval Stores Investigations - Southern Region			
S5 2-38	Preparation of chemical intermediates from pine gum products for use in the preparation of new synthetic polymers, plastics and resins to expand the utilization of turpentine and rosin.	Olustee, Fla.	Yes	11-B-1
S5 2-39 (Rev.)	Development of a method for the determination of rosin and rosin derivatives in protective coatings.	Olustee, Fla.	Yes	11-B-3
S5 2-40	Production of reactive chemical intermediates from turpentine by reaction with selected low molecular weight reagents.**	Olustee, Fla.	Yes	11-B-1
S5 2-41	Development of process for isolation of levopimaric acid from pine gum.	Olustee, Fla.	Yes	11-C-1
S5 2-42	Polyester resins from pine gum derivatives.**	Olustee, Fla.	Yes	11-B-3
S5 2-43	The preparation of polyfunctional compounds from rosin, resin acids, and resin acid derivatives through their reaction with formaldehyde.**	Olustee, Fla.	Yes	11-B-3
S5 2-44	Hypochlorite modification of rosin and resin acids for use as chemical intermediate for preparation of new industrial resins, surface coatings, plastics, rosin soap emulsifiers and similar materials.	Olustee, Fla.	No	
S5 2-45 (C)	Application of the oxo and related reactions to terpenes and resin acids to produce alcohols, aldehydes, and/or acids and the characterization of the products thus obtained.	Cincinnati, Ohio	Yes	11-B-3
S5 2-46 (C)	Synthesis of terpene alcohols and glycols for use in the production of new and useful terpene derived polymers.	Ithaca, N.Y.	Yes	11-B-3
S5 2-47	The utilization of photosensitized oxidized pine gum and components in the fields of plastics and rubber.	Olustee, Fla.	Yes	11-B-2
S5 2-48	Reaction of terpenes derived from turpentine with dienophiles to produce useful acids, aldehydes, amines, nitriles, sulfones, and related derivatives.*	Olustee, Fla.	Yes	11-B-1
S5 2-49 (C)	Investigation of the acid-catalyzed dimerization of alpha-pinene.*	Gainesville, Fla.	No	
S5 5-	New and Replacement Crops Utilization Investigations - Southern Region			
S5 5-45	Investigation on chemical modification of oils from potential new oilseed crops such as Limnanthes and Cuphea to produce materials having utility in plastics, plasticizers, synthetic rubbers, protective coatings, and other industrial products.**	New Orleans, La.	Yes	13-A-1

*Initiated during reporting year.
**Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Area & Subheading
SU-0-0-1 (DC)	Development of new and improved processed peach products, with special emphasis on the suitability of existing commercial varieties in the Southeast for the production of fresh peach concentrates.***	Griffin, Ga.	Yes	15-A-1
SU-0-0-2 (SG)	Improvement of fat emulsions suitable for use in intravenous alimentation.* ***	New Orleans, La.	Yes	6-B-1
SU P 1	Seed Protein Pioneering Research Laboratory.***	New Orleans, La.	Yes	7-A-1
SU P 2	Plant Fibers Pioneering Research Laboratory.***	New Orleans, La.	Yes	1-A-4
UR-A7-(40)-3	A study of the relationship of substituent fatty acid groups on the physical properties of diacid triglycerides of palmitic and stearic acids as a means of increasing the utilization of cottonseed oil for food and industrial purposes.	Bombay, India	Yes	6-A-3
UR-A7-(20)-4	Investigation of the photochemical degradation of cotton to derive information which would enhance the utilization of cotton.	Bombay, India	Yes	1-A-3
UR-A7-(40)-12	Effect of heat on tung oil and derivatives of tung oil and characterization and identification of compounds resulting from heat treatments, to extend the utilization of tung oil.	Poona, India	Yes	8-A-2
UR-A7-(20)-19	A study of the relation between fine structure and mechanical properties of cotton fibers by swelling and stretching treatments, as a means of improving the properties, and thereby increasing the utilization of cotton.	Ahmedabad, India	Yes	1-A-4
UR-A7-(40)-26	Studies of the addition of carbenes to unsaturated fatty materials derived from cottonseed oil, to provide possible new outlets for the utilization of cottonseed oil.	Bangalore, India	No	
UR-A7-(40)-28	Investigation of the synthesis and properties of new-type glycol mono alkyl ethers for the control of water evaporation, to extend the industrial utilization of cottonseed oil.	Poona, India	No	
UR-A7-(20)-30	Investigation of new solvents for molecular weight determination of cellulose, to obtain basic information needed to improve cotton products and thereby enhance the utilization of cotton.	Bombay, India	Yes	1-A-3
UR-A7-(20)-32	Investigation of the microbial decomposition of cellulose with special reference to the effect of Indian bacterial organisms on cotton and cotton fabrics to provide basic information for the improvement of cotton products.	Bombay, India	Yes	1-A-3
UR-A7-(20)-33	Investigation of the preparation of radioresistant and radiosensitive celluloses to obtain basic information needed for useful applications of high energy radiation in cotton textile processing, thereby enhancing the utilization of cotton.*	Bombay, India	No	
UR-A7-(20)-46	A study of the physical chemistry and thermodynamics of solution and vapor phase adsorption on and in the cotton fiber to obtain basic information needed to improve cotton processing and utilization.*	Ahmedabad, India	No	

*Initiated during reporting year.
***There are no line projects under this work project.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Area & Subheading
UR-A7-(20)-51	Investigation of means to minimize fiber hooked ends in cotton card and drawing slivers to develop processing organizations of optimum efficiency, and thus to promote increased utilization of cotton.*	Ahmedabad, India	No	
UR-A10-(20)-5	Fundamental investigation of crimp in cotton fibers and its relationship to other fiber properties, as well as its effect on processing performance and product quality.	Jerusalem, Israel	Yes	1-A-5
UR-A10-(40)-34	Investigation of π-complexed organometallic compounds derived from polyunsaturated fatty acids, to obtain fundamental information needed in expanding the utilization of cottonseed oil.	Haifa, Israel	Yes	6-A-3
UR-A10-(20)-50	A fundamental study of the oxidation of cotton and crosslinked cotton by hypochlorite, hypobromite, and other oxidizing agents, to obtain information needed on the kinetics of the oxidation and the changes in physical and chemical properties, in order to improve the characteristics of cotton for various end uses.*	Jerusalem, Israel	No	
UR-A11-(50)-7	Isolation and identification of the nucleic acid derivatives of cane molasses, in order to obtain information applicable to expanding the utilization of molasses industrially and in feeds.	Kyoto, Japan	Yes	12-A-1
UR-E9-(20)-61	A fundamental study of the relation of crystallinity to accessibility in native and modified cotton, to obtain information on the supermolecular structure of cotton that is needed in the development of improved cotton products.	Paris, France	Yes	1-A-4
UR-E10-(20)-2	Development of an apparatus for counting neps in cotton card web as an aid toward increasing the quality of cotton products.	Reutlingen-Stuttgart, West Germany	Yes	2-B-1
UR-E15-(40)-33	Investigations on the physical and physicochemical properties of cottonseed proteins, to obtain basic information needed for the increased utilization of cottonseed.	Rome, Italy	Yes	6-A-1
UR-E15-(40)-35	A study of the mechanism of gossypol toxicity counteraction by L-lysine to gain information needed to permit the increased use of cottonseed products in animal feeds.*	Milan, Italy	No	
UR-E19-(20)-4	A fundamental study of the role of the structural elements of the cotton fiber in response to stress in deformation and recovery, to obtain information needed in the development of improved cotton products.	Delft, Holland	Yes	1-A-2
UR-E19-(20)-12	An investigation of the fundamental mechanisms and bonding forces that could be used to improve the tensile strength and other physical properties of cotton textiles, as a means of increasing the utilization of cotton.*	Delft, Holland	No	
UR-E25-(20)-1	Development of methods and equipment for determining irregularity of transparency of card web and for counting of neps, by means of electronic devices, as aids to improving product quality in cotton textile operations.**	Barcelona, Spain	Yes	2-B-1
UR-E25-(20)-2	Determination of relationship between the cohesion of cotton fibers and other physical properties of fibers, rovings, and yarns, as a step in improving product quality and processing efficiency.	Barcelona, Spain	Yes	2-A-1

*Initiated during reporting year.
**Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
UR-E25-(20)-13	Determination of effect of drafting forces in high-draft systems on uniformity and strength of cotton yarns as a step in improving product quality and processing efficiency.	Barcelona, Spain	Yes	2-A-2
UR-E25-(20)-31	A study of the measurement of the "total hairiness" of cotton yarn and the determination of the mechanical factors contributing toward its formation, to obtain basic information needed to improve the processing of cotton into textiles.*	Barcelona, Spain	No	
UR-E25-(50)-36	Development of new or improved methods of synthesizing, isolating, and purifying selected terpene alcohols for use as standards, to obtain basic information on the composition and properties of products made from pine gum as an aid in developing new industrial uses for naval stores products.*	Barcelona, Spain	No	
UR-E26-(20)-2	Fundamental investigation of setting reactions for cotton fabrics and garments, to develop information basic to the improvement of cotton products, thereby increasing the utilization of cotton.	Gothenburg, Sweden	Yes	1-A-2
UR-E26-(20)-6	Basic investigation of the behavior of cotton subjected to aerodynamic forces, for the purpose of improving the processing characteristics of cotton textiles.*	Gothenburg, Sweden	No	
UR-E29-(20)-4	Fundamental investigation of the causes of warp breakage in the weaving of cotton yarns, as a basis for improving quality and reducing costs of production.**	Didsbury, Manchester, England	No	
UR-E29-(20)-6	Fundamental study of the microbiological breakdown of natural cotton fiber, as a contribution to the better preservation of cotton products.**	Didsbury, Manchester, England	No	
UR-E29-(20)-9	Fundamental study of the pyrolysis of cotton cellulose to provide information needed for improvement of flame-resistant treatments for cotton.**	Didsbury, Manchester, England	Yes	1-B-3
UR-E29-(40)-26	Studies on the fatty acid and glyceride composition of cottonseed oil and the crystallizing behavior of some of the major components, to obtain fundamental information that will contribute to the development of improved edible products and hence to expanded utilization of cottonseed oil.	Leatherhead, Surrey, England	Yes	6-A-3
UR-E29-(20)-35	Fundamental investigation of preparation and properties of esters, anhydrides, hydrazides, pseudohalides, fluorides, and related compounds of the phosphonitrilic chlorides for use in preparing new products to increase the utilization of cotton.**	London, England	No	
UR-E29-(20)-55	A fundamental study of the preparation and properties of phosphazene (phosphonitrilic) and phosphoryl chloride derivatives having potential for reaction with cotton cellulose, to obtain information needed in the development of new useful products from cotton, thus increasing its utilization.	London, England	Yes	1-B-1
UR-E29-(20)-65	A study of the effect of caustic soda and other swelling agents on the fine structure of cotton, to obtain basic information needed to improve cotton products and thereby enhance the utilization of cotton.*	Didsbury, Manchester, England	No	

*Initiated during reporting year.
**Discontinued during reporting year.

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
UR-01-(40)-2	An investigation of the chemistry and biological effects of cyclopropenoid compounds that occur in cottonseed and cottonseed products, to obtain basic information needed to improve the utilization of these commodities.*	Ryde, N.S.W., Australia	No	
UR-S9-(40)-2	Preparation, characterization, and evaluation of derivatives of gossypol from cottonseed for use as biologically active materials, ultraviolet absorbers and other valuable products.	Montevideo, Uruguay	Yes	6-D-1

*Initiated during reporting year.